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UNITED STATES DEPARTMENT OF AGRICULTURE
Bureau of Entomology and Plant Quarantine
Division of Insecticide Investigations

THE WORK OF THE DIVISION OF INSECTICIDE INVESTIGATIONS, 1927-1939

Washington, D. C.
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U. S. DEPARTMENT OF AGRICULTURE
BUREAU OF ENTOMOLOGY AND PLANT QUARANTINE

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Contents

	Page
Introduction	3
Acknowledgment	4
Insecticides of plant origin	6
Tobacco	6
Pyrethrum	8
Derris, cube, and other rotenone-containing plants	10
Derris	10
Lonchocarpus	12
Tephrosia	12
Spatholobus	13
Parosela (family Fabaceae)	13
Quassia and Aeschrion (family Simaroubaceae)	14
Croton (family Euphorbiaceae)	14
Haplophyton (family Apocynaceae)	14
Phellodendron (family Rutaceae)	14
Helenium (family Compositae)	14
Anamirta (family Menispernaceae)	15
Anabasis (family Chenopodiaceae)	15
Miscellaneous insecticidal plants	15
Synthetic organic insecticides	15
Pyridine and pyrrolidine derivatives	15
Thiocyanates	16
Halogenated organic compounds	16
Dinitrophenol and derivatives	16
Azo compounds	17
Heterocyclic compounds containing nitrogen and/or sulfur	17
Phenothiazine	17
Phenothioxin (phenoxathiin)	17
Dimethylacridan	18
Phenazine	18
Xanthone	18
Miscellaneous synthetic compounds	18
Relationship between toxicity and chemical constitution	19

Page
Inorganic insecticides 20
Lead arsenate 20
Calcium arsenate 20
Paris green and homologs 21
Other Arsenicals 22
Fluorides 22
Mercuric chloride 23
Copper compounds 23
Sulfur 23
Spray residues 24
Fumigants 26
Formates 28
Ethylene dichloride and propylene dichloride 28
Ethylene oxide 28
Chloropicrin 29
Hydrogen cyanide 29
Chlorine 30
Accessory materials 30
Wetters and spreaders 30
Diluents 31
Stickers 31
Emulsifiers 31
Analytical investigations 31
Application of statistical methods to insecticide problems 34
New and improved apparatus 35
Chemotropism 36
Pharmacological investigations 37
Mothproofing 37
Tree medication 38
Economics of insecticides 38
Bibliographies, abstracts and reviews 38
Abstracts of patent literature 39
Book reviews 40
Popular articles on insecticides 40
Papers prepared by invitation 40

INTRODUCTION

On July 1, 1927, two of the bureaus in the United States Department of Agriculture, namely, the Bureau of Chemistry and the Bureau of Soils, were consolidated to form the Bureau of Chemistry and Soils (now known as the Bureau of Agricultural Chemistry and Engineering), and at the same time the Division of Insecticide Investigations was created as one of the units of the new bureau for the purpose of prosecuting chemical research on insecticides and fungicides. On September 1, 1934, this Division was transferred to the Bureau of Entomology and Plant Quarantine, and has remained in this Bureau since that time.

The authority for the work of this Division is contained in the various appropriation bills for the Department of Agriculture and reads in part as follows:

> "Insecticide and fungicide investigations: For the investigation and development of methods of manufacturing insecticides and fungicides, and for investigating chemical problems relating to the composition, action, and application of insecticides and fungicides."

The organization and research line projects of the Division of Insecticide Investigations as of December 31, 1939, are shown in the chart which follows page 2.

During the 12 1/2-year period ended December 31, 1939, the Division of Insecticide Investigations has published 480 papers, and its members have been granted 60 patents. Other papers and patent applications are pending, as shown in the following tabulation:

Status of publication, December 31, 1939

Papers published	480
Papers approved, but not yet published	19
Papers submitted, but not yet approved	19
Total	518
Patents granted	60
Patent applications in interference	2
Patent applications pending	36
Total	98

The appropriation allotted the Division has ranged from $34,800 for the fiscal year 1928 to $161,269 for each of the fiscal years 1937 and 1938. A detailed report is as follows:

Appropriations

Fiscal year	Total appropriation
1928	$ 34,800
1929	60,300
1930	101,765
1931	118,000
1932	128,400

1933	117,300
1934	93,000
1935	132,352
1936	148,984
1937	161,269
1938	161,269
1939	143,969
1940	152,569

The following account of accomplishments has been made as brief as is compatible with clarity. The numbers in parentheses are the index numbers of the publications of the Division, a complete list of which is appended to this report. United States patents granted members of the Division are referred to under their own numbers. Full details of all research work done in the Division will be found in these publications and patents.

ACKNOWLEDGMENT

It should be emphasized that the work of the Division of Insecticide Investigations is wholly physical and chemical and that it makes no tests upon insects, either in the laboratory or in the field. All tests of materials to determine their insecticidal value referred to in this report were made by members of other divisions of the Bureau of Entomology and Plant Quarantine. Furthermore, many of the purely chemical studies, especially of analytical methods and of processes of washing apples, have been carried out in cooperation with chemists, horticulturists, and others in the Bureau of Agricultural Chemistry and Engineering, the Food and Drug Administration, the Bureau of Plant Industry, and certain State agricultural experiment stations. Much of the work of the Division of Insecticide Investigations has been cooperative, as shown by the fact that 107 out of 480 published papers and 3 out of 60 patents represent the joint efforts of our workers with those in other organizations.

A summary of this cooperative work is indicated below. The names of the cooperating entomologists and other scientists are also indicated by asterisks in the list of publications at the end of this report.

Cooperative work with various organizations, as shown by number of publications and patents issued jointly with each worker.

United States Department of Agriculture.

Bureau of Entomology and Plant Quarantine:-

Division of Insects Affecting Man and Animals:

	Number of publications
Back	2
Bishopp	3
Laake	3
Parman	3

DEPARTMENT OF AGRICULTURE

BUREAU OF ENTOMOLOGY AND PLANT QUARANTINE

DIVISION OF INSECTICIDE INVESTIGATIONS

ORGANIZATION CHART

JAN. 1, 1940

R.C. ROARK - IN CHARGE P-8
C.M. SMITH - ACTING IN CHARGE P-5
S.R. Gersten CAF-5
S.A. Davidson CAF-3
R.O. Ericson SP-4
M. Iverson SP-1
M.L. Allen Cu-2

INSECTICIDAL PLANTS

H.L. Haller P-6
Project Leader
Miscellaneous Plants
E.P. Clark P-5
M.S. Schechter P-2
J.P. Hewlett SP-4
Pyrethrum
F.B. LaForge P-5
F. Acree P-2
Tobacco, etc.
C.R. Smith P-4
L.N. Markwood P-4
Rotenone bearing plants
L.D. Goodhue P-3
J.W. Wood SP-4
Geraniol
H.A. Jones P-3
J.W. Wood SP-4

SYNTHETIC ORGANIC INSECTICIDES

H.L. Haller P-6
Project Leader
Organic Compounds in general.
L.E. Smith P-4
C.V. Bowen P-2
S.I. Gertler P-2
M.S. Schechter P-2
Phenothiazine
L.E. Smith P-4

INSECTICIDAL DEPOSITS

C.M. Smith P-5
Project Leader
Miscellaneous
R.H. Carter P-3
Arsenicals
R.H. Carter P-3
C.W. Murray P-3
H.D. Mann SP-4
Fluorides
R.H. Carter P-3
Nicotine
L.N. Markwood P-4

INORGANIC INSECTICIDES

C.M. Smith P-5
Project Leader
Calcium Arsenate
O.A. Nelson P-4
Sodium Arsenite
O.A. Nelson P-4
White Arsenic
F.E. Dearborn P-3

FUMIGANTS

C.M. Smith P-5
Project Leader
Heat-dispersion of relatively Non-volatile Insecticides
L.D. Goodhue P-3
J.W. Wood SP-4

ACCESSORY MATERIALS

C.M. Smith P-5
Project Leader
Spreaders, Wetters, etc.
H.L. Cupples P-4
Adhesives for Organic Compounds
H.L. Cupples P-4

TOXICITY STUDIES

C.M. Smith P-5
Project Leader
Tests on Goldfish
W.A. Gersdorff P-3

ANALYTICAL INVESTIGATIONS

C.M. Smith P-5
Project Leader
Analytical Service
R.H. Carter P-3
C.W. Murray P-3
D.L. Vivian P-2
H.D. Mann SP-4
Chemical Methods
R.H. Carter P-3
C.W. Murray P-3
D.L. Vivian P-2
Physical Methods
E.L. Gooden P-1

FIELD LABORATORIES

MOORESTOWN, N.J.

R.D. Chisholm P-4
In charge of laboratory
Insecticides for Japanese Beetle
R.D. Chisholm P-4
Chemical Estimation of Effectiveness of Treated Soil
R.D. Chisholm P-4
L. Koblitsky P-1
Analysis of Control Samples
L. Koblitsky P-1
R.W. Coles SP-3
J.J. Lockowitz SP-3
Analytical Methods
R.D. Chisholm P-4
L. Koblitsky P-1

VINCENNES, IND.

J.E. Fahey P-3
In charge of laboratory
Insecticidal Deposits
J.E. Fahey P-3
J.F. Cassidy P-1
H.W. Rusk SP-4
Insecticides for Codling Moth
J.E. Fahey P-3
J.F. Cassidy P-1
H.W. Rusk SP-4

MANHATTAN, KAN.

H.D. Young P-3
In charge of laboratory
Fumigation of Stored Grain and Cereal Products
H.D. Young P-3

WHITTIER, CALIF.

R.A. Fulton P-4
In charge of laboratory
Fumigation of Calif. Red Scale
R.A. Fulton P-4
R.L. Busbey P-2
Sulfur
R.A. Fulton P-4
R.L. Busbey P-2

YAKIMA, WASH.

C.C. Cassil P-3
In charge of laboratory
Insecticidal Deposits
C.C. Cassil P-3
Insecticides for Codling Moth
C.C. Cassil P-3

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	Number of Publications
Division of Control Investigations:	
Bulger	1
Campbell	7
Fink	5
McGovran	1
McIndoo	2
Phillips	1
Richardson, C. H.	1
Richardson, H. H.	1
Shepard	2
Sullivan	12
Division of Cereal and Forage Insect Investigations:	
Cotton	17 (+ 3 patents)
Wagner	8
Division of Fruit Insect Investigations:	
Cressman	3
Haas	1
Lathrop	1
Munger	6
Newcomer	4
Sazama	7
Siegler	8
Steiner	8
Summerland	1
Yothers	1
Yust	2
Division of Truck Crop and Garden Insect Investigations:	
Davis	4
Weigel	1
Bureau of Plant Industry:	
Diehl	3
Haller, M. H.	4
Leukel	2
Reeves	1
Ryall	5
Smith, Edwin	3
Food and Drug Administration:	
Abbott	1
Clifford	1
Davidson, W. M.	4
Graham	2
Harris	1
Hillig	2
Keenan	5
Loughrey	1
McDonnell	1
Vorhes	1
Wichmann	2

	Number of publications
Bureau of Agricultural Chemistry and Engineering:	
Davidson, J.	1
Palkin	1
Pulley	1
Machine Shops:	
Swenson, R. L.	1
Bureau of Agricultural Economics:	
Cox	1
Maryland Agricultural Experiment Station:	
Beaumont	3
University of Maryland:	
Haring	1
West Virginia Agricultural Experiment Station:	
Gould	2

INSECTICIDES OF PLANT ORIGIN

Beginning in 1927, research has been prosecuted on the principal insecticides of plant origin such as nicotine, anabasine, rotenone, deguelin and related rotenoids, quassin, and pyrethrins I and II. Exploratory work has been carried on with many plants, including Nicotiana, Pyrethrum, Derris, Lonchocarpus, Tephrosia, Spatholobus, Parosela, Quassia, Aeschrion, Croton, Haplophyton, Phellodendron, Helenium, Anamirta, and others in an effort to find new compounds of insecticidal value.

The results of this work are discussed under separate headings.

TOBACCO

Tobacco (Nicotiana tabacum L., family Solanaceae) is the only source of the widely used and potent insecticide nicotine. About 1,000,000 pounds of this alkaloid are consumed annually in the United States, mostly in the form of an aqueous solution of the sulfate containing 40 percent of nicotine. Nicotine is not only a contact insecticide and a fumigant, but when made into certain water-insoluble forms it is also a valuable stomach poison for some insects. The Division of Insecticide Investigations has been active in the search for new nicotine compounds and new and better ways of applying nicotine against pests. New uses for nicotine help farmers, orchardists, florists, and poultry raisers to better control of their insect enemies and also widen the market for surplus and off-grade tobacco, thus aiding the tobacco grower. The United States, during the 3-year period 1935-37, produced an average crop of tobacco amounting to 1,335,296,000 pounds, or about 23 percent of the world production. The estimated farm value averaged nearly 277 million dollars. Because of reduction in exports of fire-cured and dark air-cured tobaccos, unsalable surpluses have developed since 1929. Of means proposed to reduce these surpluses an increased

consumption of tobacco as a source of nicotine is the most promising, and the Division of Insecticide Investigations has contributed notably to this end.

Our accomplishments in these endeavors include the development of the new nicotine compounds nicotine peat (286, 351, 410, and U. S. Patent 2,107,058) and nicotine humate (287 and U. S. Patent 2,066,941) by Markwood, and nicotine bentonite (222 and U. S. Patent 2,096,566) by C. R. Smith. The latter has given promising results in orchard tests in Indiana (Steiner and associates, and Fahey, [illegible], 384, 405, 447) and elsewhere as a substitute for lead arsenate for codling moth control.

The removal from apples of nicotine-bentonite spray residues was investigated at Vincennes, Ind., by Fahey, Rusk, Steiner, and Sazama (407) and at Yakima and Wenatchee, Wash., by Cassidy of this Bureau and Edwin Smith of the Bureau of Plant Industry (465). Either hydrochloric acid or sodium silicate washing solution is effective in removing these residues.

The increased use of nicotine (and, in turn, of tobacco) due to the development of this new nicotine compound for codling moth control may be seen from the following figures of the recent consumption of nicotine in the apple belt of Indiana and Illinois.

Consumption of nicotine for codling moth control in Indiana and Illinois

Year	Pounds (of alkaloid)
1936	400
1937	4,000
1938	40,000
1939	80,000

The possibilities of a still greater use of nicotine as a substitute for lead arsenate as a poison for codling moth control are apparent when it is realized that the greater part of the annual consumption of about 40 million pounds of this arsenical in the United States is applied in apple orchards.

The Secretary of Agriculture (Senate Document No. 39, 76th Congress, 1st Session) in a letter dated March 17, 1939, transmitting in response to Senate Resolution No. 291 (75th Congress) a report pertaining to the export trade in and byproducts uses of tobacco, wrote as follows:

> "Nicotine bentonite used as a poison for chewing insects establishes a decided step forward in insecticide science. Its use is believed to involve no objectionable residue problem as is the case with arsenate of lead, now the most widely used poison in the horticultural field. A further advantage in the use of this material is that it does not damage the foliage of the plants upon which it is employed nor is there any accumulation of poison in the soil. This material has passed the experimental stage and in certain fruit areas it has been adopted in commercial practice. *** It is estimated that if the present indicated rate of increase in nicotine usage for the control of the codling

moth is maintained, 1 million pounds of nicotine alkaloid might be used annually by the apple-growing industry alone within 10 years. Such new demand for nicotine would absorb the available supplies of the low grades in the heavy types of tobacco, and would require very much larger amounts of low grades of the lighter types such as flue-cured and burley tobaccos than are now being consumed. Such new demands would be in addition to the 12 to 15 million pounds of low-grade leaf tobacco and approximately 120 million pounds of factory wastes now used annually for contact spray purposes."

It was observed by C. R. Smith (U. S. patent 2,033,856) that bentonite combines with other alkaloids and natural bases, and Markwood (U. S. patent 2,123,248) found that peat does also. These compounds may prove useful as new modes of administering drugs such as arecoline, strychnine, etc., to domestic animals and man where a delayed action is desired.

New analytical methods for nicotine were perfected by Spies (317, 320), by Goodhue (374), and by Markwood (378) which make it possible to determine quickly and accurately the nicotine spray residue on a single apple, or the minute quantity of nicotine vapor present in the atmosphere of a greenhouse that has been fumigated with this material. These data must be known to the entomologist in order that an effective and economical schedule for the application of nicotine can be worked out.

In connection with our studies on tobacco, other species of the genus Nicotiana and hybrids of these with tobacco have been examined for constituents of insecticidal value. In addition to nicotine, C. R. Smith isolated anabasine (249) and laevo-nornicotine (356) from certain of these plants, and the plant breeder has been given information which enables him to produce a hybrid rich in any one of these alkaloids. This work has value not only to those seeking improved tobacco varieties, but also to those who wish to develop new and improved sources of insecticides. For example, anabasine is at present obtained commercially only from the Russian weed Anabasis aphylla L. (family Chenopodiaceae), and the finding of a domestic source of this valuable insecticide in Nicotiana glauca will free the United States from a foreign monopoly.

New nicotine compounds made by the Division of Insecticide Investigations include its salts with alpha-bromo-palmitic and alpha-bromo-stearic acids. These salts are more soluble in oil than in water and hence, when added to an oil-in-water emulsion, tend to remain in the oil phase, whereas nicotine sulfate and other nicotine salts and also free nicotine go largely into the aqueous phase and are lost when the emulsion breaks on contact with foliage and the water part of the emulsion runs off.

Directions were published for preparing home-made nicotine solutions from tobacco (269). There were also issued bibliographies of the chemistry and of the insecticidal uses (306, 319 by McIndoo and Busbey) of nicotine and tobacco. These present 3,348 abstracts of published articles and comprise the most complete information on the subject that has been published.

PYRETHRUM

The flowers of certain species of chrysanthemum (Chrysanthemum

cinerariaefolium (Trev.) Vis., family Compositae) have long been known to have insecticidal value. Under the name pyrethrum as much as 20,000,000 pounds of these flowers have been imported into the United States in a single year for use in the manufacture of household and cattle fly sprays, greenhouse sprays, and agricultural dusts. The chemist is interested in learning the exact make-up of the two principles that are responsible for the insecticidal action of these flowers. These principles, called pyrethrin I and pyrethrin II, look like glycerol but have very complex chemical structures which, if completely understood, would enable the chemist to synthesize related materials of perhaps analogous action. A knowledge of the structures of the pyrethrins is also necessary in order to determine their compatibility with other insecticides and to make it possible to use them most effectively and economically.

LaForge and Haller developed an improved procedure for the isolation and partial separation of pyrethrin I and pyrethrin II (265, U. S. patents 2,044,502 and 2,050,974), which, in careful laboratory tests by Sullivan, McGovran, and Phillips (406), have been shown to act differently on flies, the knockdown being due to pyrethrin II and the kill to pyrethrin I. The structures of these principles, although not wholly determined, have been revised by Haller, LaForge and Acree (263, 282, 291, 307, 340, 341, 355, 370, 399, 430, 443, 478), and a new constituent of pyrethrum flowers, called chrysanthin, was isolated and studied (382, 453) and found to be identical with the pyrethrosin of Thoms. The behavior of the pyrethrins on hydrogenation was studied by Haller and LaForge (340), and the lack of insecticidal value for flies of the resulting products ascertained by Sullivan (385). One of the reduction products is an odorous compound used in the perfume industry, namely, dihydrojasmone (Haller and LaForge U. S. patent 2,096,715). In the course of this work it was shown that crystalline pyrethrin I semicarbazone is not a homogeneous compound (328, 329), and pyrethrin II was isolated pure for the first time in the form of its crystalline semicarbazone.

It was found that the common field daisy contains no pyrethrins (Acree and LaForge, 302). This weed resembles pyrethrum closely and has been used to adulterate the genuine flower.

A new method of determining pyrethrin II was devised by Haller and Acree (263) which is quicker and more accurate than previous methods. The pyrethrin content of fresh flowers was determined (297, 326). The value of ethylene dichloride for extracting the insecticidal principles from pyrethrum flowers was ascertained by Gersdorff of this Bureau, and Davidson of the Food and Drug Administration (43), and, following an announcement of our findings, this solvent came into large-scale use in the manufacture of concentrated pyrethrum extracts. A review of assay methods for pyrethrum flowers was published by Roark (25); attention was called by the same author to the incompatibility of pyrethrum and alkaline soaps (58), and aqueous extraction of pyrethrum was found by Schechter and Haller (413) to be unsuitable for increasing the percentage of pyrethrins in the flowers and in the petroleum ether extracts.

In cooperation with McDonnell and associates of the Food and Drug Administration (66), a report on the relative insecticidal value of commercial grades of pyrethrum was published by Nelson. This called attention for the first time to the fact that open flowers are more valuable than

closed flowers, a finding directly contradictory to the opinion of the trade.

An examination by Gertler and Haller (425) of methods recommended for preparing home-made pyrethrum-kerosene fly sprays showed that, although the pyrethrins are readily dissolved in the kerosene employed, there is a loss of 27 percent in the volume of the solution when 1 pound of flowers is macerated in 1 gallon of kerosene and the extract decanted. In order to obtain a solution containing 100 mg. total pyrethrins per 100 cc. (the minimum standard for a satisfactory fly spray) it is often necessary to use more than 1 pound of flowers in order to make 1 gallon of spray because of the low quality of much of the pyrethrum on the American market. In the case of pyrethrum powder containing only 0.45 percent total pyrethrins -- and material of even lower grade has been encountered -- it is necessary to use about 3-1/2 pounds of pyrethrum powder and 1-3/4 gallons of kerosene in order to obtain 1 gallon of minimum standard fly spray.

DERRIS, CUBE, AND OTHER ROTENONE-CONTAINING PLANTS

Derris. -- In 1927 the Division of Insecticide Investigations began an investigation of derris. At that time Japanese chemists were investigating one of the most important constituents of derris, namely, rotenone, but its structure was unknown, and the presence in derris of deguelin, tephrosin, and toxicarol was unsuspected.

Work on rotenone resulted in the determination of its chemical structure in November 1931, and an account of this was published in February 1932. This achievement was recognized in 1933 by the award of the Hillebrand Prize of the Washington Chemical Society to Doctors LaForge and Haller, who, with the assistance of L. E. Smith, were the first to solve this very difficult problem. During the course of this work our chemists were in keen competition with chemists in Japan, Germany, and England who were also seeking to determine the structure of rotenone. Accounts of this research were published in a series of 32 papers by LaForge, Haller, and L. E. Smith (35, 51, 52, 53, 61, 65, 69, 74, 82, 83, 93, 109, 115, 117, 120, 124, 125, 126, 129, 137, 138, 150, 152, 160, 190, 191, 193, 199, 220, 223, 231, and 232) and also a general review of all the work done to determine the structure of rotenone (179).

At the same time the other constituents of derris and cube were studied by Clark (56), namely, deguelin (87, 89, 104, 111, 118, 151, 163), tephrosin (92, 155, 171), and toxicarol (60, 108, 136, 144, 163, 214). Of these deguelin is the most toxic to insects, and the optically active form is more potent than the optically inactive form (Fink and Haller, 296).

Racemic deguelin was found by Goodhue and Haller (436) to form definite stable solvates with a number of solvents. Deguelin crystallizes from carbon tetrachloride, chloroform, bromoform, and ethylene bromide with 1 mole of solvent to 1 mole of deguelin, and from bromobenzene, chlorobenzene, and benzaldehyde with 1 mole of solvent to 2 moles of deguelin. The optically active deguelin in the roots of Derris and Lonchocarpus does not form these solvates. A method for the quantitative determination of deguelin in plant material based on the formation of the carbon tetrachloride solvate has been published (479). Seven samples of derris contained from 0.35 to 3.9 percent deguelin and 6 samples of Lonchocarpus from 0.25 to 2.3 percent.

The physical properties of rotenone were investigated, such as its dimorphism (Gooden and C. M. Smith, 274) and its solubility in many organic solvents (Jones et al. 62, 366) with some of which it forms solvates (113, 332). This property is the basis of the assay method now used for evaluating derris and cube. The combination of rotenone with dichloroacetic acid was patented by Jones (U. S. patent 2,103,195).

The milky sap from fresh derris root has been used as an insecticide for centuries by Chinese gardeners, but in those parts of the world where only the dried root is available little attention has been given to water extracts. Goodhue and Haller (430) reported a chemical examination of the water extracts of seven samples of derris, cube, and timbo. From 24 to 41 percent of the active material can be removed by one extraction, and successive extractions remove more. When preserved against fermentation, these suspensions of the active principle are very stable. Both glucose and levulose were found to be present in one sample. The glucosides similar to saponin had no hemolytic power.

The value of rotenone as a mothproofing agent was discovered by Roark in cooperative studies with Back and Cotton (85), and its toxicity to goldfish was determined by Gersdorff (86). Some ways of preparing rotenone for use as an insecticide were looked into by Jones in cooperation with Davidson of the Food and Drug Administration (95, 96), and certain procedures were patented (1,928,256; 1,928,968). The value of kerosene extracts of derris as fly sprays was pointed out by Campbell, Sullivan and Jones (210, 215). The use of carbon tetrachloride for extracting rotenone from plant material was patented by Jones (1,942,104); also a process of making dihydrorotenone from rotenone by the use of a nickel catalyst was patented by Haller and Schaffer (193, 199, and U. S. patent 1,945,312).

Attention was called by Roark to the absence of rotenone in certain shipments of commercial derris root (98) received in 1930, and the availability of derris of high (9 to 12 percent) rotenone content on the American market in 1938 was pointed out by Jones (441). Both the Federated Malay States and the Dutch East Indies can supply such roots in commercial quantities.

The rate and extent of decomposition of rotenone under different conditions was investigated by chemical analysis (101, 110) and also in cooperation with Davidson (96) and Campbell and Sullivan (177) by tests upon insects; and the stability of derris spray residues under different conditions of temperature, light, and rainfall was studied (420, 442) by spraying several derris mixtures on glass plates and determining the rotenone in the deposits after exposure under various conditions of temperature and light. Derris deposits were decomposed both in the presence and in the absence of sunlight. The rate of decomposition was increased when the temperature was increased. After exposure for 15 days to sunlight at 32.5° F., an average of 60 percent of the deposit was decomposed, while 73 percent was decomposed at 70-75° F., even though the latter deposits were subject to the screening effect of greenhouse glass. In the absence of light and at similar temperatures there was no measurable change. However, when the temperature was increased to 120° F., about one-fourth of the deposit was decomposed.

The history of the use of derris as an insecticide from 1848 through 1928 has been compiled by Roark (421, 434), and its later history is being

prepared. A list of nearly 50 species of insects, belonging to 5 orders and 26 families, that attack stored derris root or the leaves of the growing plant (438) and a list of the native names for various species of Derris (448) were published by the same author.

Lonchocarpus. -- In the course of investigating miscellaneous fish-poison plants, it was found that rotenone and deguelin are also present in the South American plant cube (Lonchocarpus sp.) (Clark 41, 56; Jones 164, 202).

A new compound, lonchocarpic acid, was isolated by Jones from a species of Lonchocarpus from Venezuela (217), and it was found by the same investigator that the rotenone content of stored cube root attacked by borers (Dinoderus) was not diminished (380).

Tephrosia. -- Rotenone was discovered in a plant native to the United States, namely, Tephrosia (Cracca) virginiana, or devil's shoestring (Clark, 175). The importance of this plant as a possible source of rotenone and related insecticides was pointed out by Roark (221) and led to an extensive investigation, still under way, of this genus by the Bureau of Plant Industry. Rotenone or related compounds were later found to be present in 5 other domestic species of this genus, and their insecticidal possibilities were studied by Jones, Campbell, and Sullivan (264). A South American species of this genus, namely, Tephrosia toxicaria, was tested for insecticidal value by Jones and Sullivan (352), and toxicarol was first isolated from this species by Clark (60).

The similarity in chemical composition and insecticidal action as well as in botanical characteristics of certain genera of Fabaceae, notably Derris, Lonchocarpus, and Tephrosia, was pointed out by Roark (187). Bibliographies on the rotenone-bearing plants Derris (134), Lonchocarpus (279, 419), and Tephrosia (327) were published by the same author; also reviews and popular articles on them and rotenone (28, 31, 42, 76, 77, 133, 238, 244, 414).

A careful study by Jones, Campbell, and Sullivan (245) was made of the relations between chemical composition and insecticidal effectiveness of rotenone-bearing plants. Acetone extracts of derris, cube, haiari, and Tephrosia virginiana were prepared and tested on houseflies. The amounts of rotenone present in the samples were too low to account for all the toxicity. In more than half the samples the figures by the Gross and Smith test, considered as representing the sum of rotenone and deguelin, agreed with the toxicity value, but in other samples they were lower. Total-extractive values were higher than toxicity, and values based on the methoxyl content of the extract were somewhat closer but were also too high. When an approximate value for toxicarol was subtracted from the methoxyl figures, the results agreed more closely with the toxicity figures in general than did the results of other determinations. It was concluded that no one chemical determination accurately measures the insecticidal effectiveness of rotenone-bearing plants.

Numerous qualitative color tests have been proposed for rotenone and other constituents of derris and cube roots, and several quantitative color reactions have been developed for the evaluation of these materials. Jones (459) in 1939 compared the results obtained by some of these colorimetric

methods with each other and with some of the gravimetric methods. He concluded that in evaluating derris and cube roots it is now possible, by making use of colorimetric procedures and a determination of rotenone by the usual method, to obtain at least approximate values for deguelin and toxicarol; by the Goodhue modification of the Gross and Smith color test, to arrive at an estimate of the insecticidal value (for houseflies); and by the Meyer color test, to get a rough idea of the total materials of the rotenone type.

Cube, timbo, and derris roots yielded small quantities (0.02 to 0.40 percent) of alkaloidal material which, at a concentration of 1:10,000, was nontoxic to mosquito larvae (Sullivan) and hence was not further investigated (Jones, 464).

A study of toxicity to the housefly of optically active and inactive compounds of the rotenone series was reported in 1939 by Sullivan, Goodhue, and Haller (457). The toxicity tests were made on the housefly by the turntable method of Campbell and Sullivan (Soap 14 (6): 119-125, 149. 1938). The compounds were applied both in acetone and in a mixture of a highly refined kerosene and cyclohexanone (90:10). The more dilute acetone sprays were prepared by adding acetone to the original solution, and the more dilute kerosene-cyclohexanone sprays by adding refined kerosene. Six tests with about 150 flies each were made on each concentration of the various materials. Rotenone, levo-dihydrorotenone, levo-beta-dihydrorotenone, levo-dihydrodeguelin, levodeguelin concentrate, racemic deguelin, and racemic dihydrodeguelin were tested. In acetone solution the two optically inactive (racemic) compounds are much less toxic than are the optically active ones, but when tested in highly refined kerosene containing cyclohexanone the toxicity of the racemic compounds is approximately the same or only slightly less than that of the optically active compounds.

At present more than 3,000,000 pounds of rotenone-bearing roots are imported annually into the United States, from which are manufactured agricultural dusts for combating cabbage worms, the Mexican bean beetle, the pea aphid, the pea weevil, and other insects attacking truck crops and gardens. Rotenone and related compounds (deguelin, etc.) also go into fly sprays, cattle sprays, and horticultural sprays for combating many household and greenhouse insects.

The value of rotenone as an insecticide is due to its extraordinary potency (as a stomach poison to silkworms it is 15 times as toxic as lead arsenate, and to bean aphids it is 30 times as toxic as nicotine) together with its low toxicity to mammals (only one-thirtieth as toxic as lead arsenate when fed to rabbits). Deguelin also has high insecticidal value and adds to the effect of rotenone in derris, Lonchocarpus, and Tephrosia.

Spatholobus. -- Rotenone was demonstrated to be present in a species of Spatholobus (Jones, 176). All plants in which rotenone has been found belong to the bean family (Fabaceae).

PAROSELA (Family Fabaceae)

Two constituents were isolated by Spies and Drake (149) from the Florida plant Parosela barbata (Oerst.) Rydb., but proved nontoxic to insects.

QUASSIA and AESCHRION (Family Simaroubaceae)

Surinam quassia is derived from Quassia amara L. and Jamaica quassia from Aeschrion excelsa (Swartz.) Kuntze. Both these species have been studied chemically by Clark (333, 365, 389) and the nature of their active principles clarified. About 1,000,000 pounds of quassia wood are imported annually, but as an insecticide its use is restricted largely to the control of the hop aphid. A better knowledge of the chemistry of this material will enable the entomologist to extend the range of its usefulness. A bibliography of quassia was published (Busbey, 449).

CROTON (Family Euphorbiaceae)

The use by the Chinese of plants of this genus as fish poisons has been recorded (Roark, 112). The beans of Croton tiglium L., the source of croton oil, have been used by the Chinese as an insecticide also. A chemical study of the constituents of these beans by Spies (234, 235, 236, 342) resulted in the discovery of an analogous relationship between croton resin and the active constituent of poison ivy. The presence of a glycoside containing d-ribose in croton bean was confirmed by Spies and Drake (243). Tests upon goldfish showed croton resin to be even more toxic than rotenone, which at a concentration of only 0.04 mg. per liter (1 part in 25,000,000 parts of water) kills goldfish in 200 minutes. Croton resin would doubtless be a valuable addition to our list of potent organic insecticides if its strong vesicant action (similar to that of poison ivy) did not preclude its practical use.

HAPLOPHYTON (Family Apocynaceae)

The Mexican plant Haplophyton cimicidum A. DC. was studied, but unexpected difficulty was encountered in isolating any constituent of insecticidal value. Mannitol (Drake and Spies, 75) and quebrachite (Clark, 290), which have no insecticidal value, were identified as constituents of the plant. In Mexico the plant is used for combating cockroaches and other household insects. It is planned to reexamine this plant.

PHELLODENDRON (Family Rutaceae)

The fruit of the Amur cork tree, Phellodendron amurense, was found to be toxic to codling moth larvae and mosquito larvae in laboratory tests made by entomologists of the Bureau. A chemical study of this plant is being continued. The material is supplied by the Soil Conservation Service of the Department, which is interested in growing the tree to prevent soil erosion.

HELENIUM (Family Compositae)

The common sneezeweed of the Southern States known as Helenium autumnale L. has been reported to have some insecticidal value. A very bitter compound, helenalin, was isolated from the plant (Clark, 310) and studied in the hope that it might prove an acceptable substitute for the quassin of quassia wood, our supply of which is all imported. Studies on several species of Helenium are being continued, from three of which a new bitter compound, toxic to fish, called tenulin (Clark, 456) has been extracted.

ANAMIRTA (Family Menispernaceae)

Picrotoxin, a constituent of fish berries, Anamirta cocculus (L.) Wight & Arn., was studied and an improved method for its extraction described by Clark (255). Picrotoxin has been used in certain proprietary insecticides and its possibilities for insect control deserve careful investigation.

ANABASIS (Family Chenopodiaceae)

Attention was called to the work of Russian chemists in isolating anabasine from the plant Anabasis aphylla L. and to the identity of the product neonicotine synthesized from pyridine and this alkaloid (C. R. Smith, 121, 127).

The physical characteristics of natural anabasine were determined for the first time by Nelson (229).

Tests upon mosquito larvae by Campbell, Sullivan, and C. R. Smith (178) showed anabasine to be less toxic than nicotine, but tests by others upon aphids showed anabasine to be superior. Later, anabasine was found by us in the southwestern American plant Nicotiana glauca R. Grah. (C. R. Smith, 249), and it may be possible to develop a commercial supply of this valuable insecticide from what is now a worthless weed.

MISCELLANEOUS INSECTICIDAL PLANTS

Lists of plants reputed to have insecticidal value have been compiled by Roark, for the guidance of workers in this field, in cooperation with Keenan of the Food and Drug Administration (94,112). Tests of certain plant extracts against goldfish were reported by Spies and Drake (130, 173).

SYNTHETIC ORGANIC INSECTICIDES

Except for a few compounds used as fumigants, organic materials of insecticidal value were, until recently, all of natural origin. Examples are nicotine, the pyrethrins, rotenone, petroleum oils, and soaps. Of these, only nicotine has been synthesized, and the resulting product, being a racemic mixture, is less active than the naturally occurring laevo-nicotine and is, moreover, much more costly. Balked in his endeavor to synthesize these organic insecticides, the chemist has prepared compounds of simpler structure but which contain some grouping of atoms found in the more complex natural product. For example, the nicotine molecule is made up of a pyridine ring and a pyrrolidine ring. Many derivatives of these two compounds have been made and studied for insecticidal action but only one approaches nicotine in toxicity.

PYRIDINE AND PYRROLIDINE DERIVATIVES

Among the derivatives made by the Division of Insecticide Investigations are the dipiperidyls (13) and the dipyridyls (47) made by C. R. Smith and the three benzylpyridines (17) and various derivatives of pyrrolidine (18) and of pyridine (19) made by LaForge. Richardson and Shepard found that of these compounds the only one of outstanding insecticidal value is beta-pyridyl-alpha-piperidine reported by C. R. Smith (33, 80, 88), to which the name neonicotine was given. Later the optically active form of this

alkaloid was isolated from a weed belonging to the sugar-beet family (Chenopodiaceae) by Russian chemists and called by them anabasine from the name of the plant, Anabasis aphylla L. (121, 127).

THIOCYANATES

The high insecticidal value of the vapors of the readily volatile organic thiocyanates was first discovered in the United States Department of Agriculture (Neifert et al., U. S. Dept. Agr. Dept. Bul. 1313, 1925). Later, one of these, namely, methyl thiocyanate, was found, by Lathrop, Yust, and Cupples (248, 298), to be very destructive to the California red scale when used as a fumigant, but it proved to be too injurious to citrus trees for practical use. Following this lead new thiocyanates and also isothiocyanates were synthesized (148, 318) and found to be highly toxic to goldfish. There are now marketed as insecticides in the United States lauryl thiocyanate (Loro), various thiocyano derivatives of ethylene glycol, diethylene glycol and their esters and ethers (Lethanes), and also naphthyl isothiocyanate (Kesscocide).

HALOGENATED ORGANIC COMPOUNDS

Many compounds belonging to this class find extensive use in pest control. Examples are carbon tetrachloride, ethylene dichloride, propylene dichloride, p-dichlorobenzene, and chloropicrin.

A study of organic fumigants by Roark and Cotton (54) led to the generalization that iodo-compounds are more toxic than bromo-compounds and these in turn are more toxic than chloro-compounds. Of several halogenated compounds that were synthesized, two were deemed sufficiently insecticidal to justify patenting them for this use. These two compounds are iodonitrobenzene (U. S. patent 2,110,493, L. E. Smith and Claborn) and pentaerythrityl bromide (U. S. patent 2,140,431, Rose and Haller). These materials are believed to have great possibilities for insect control.

Halogenated azo compounds have also been patented for insecticidal use by Vivian and Haller (U. S. patents 2,095,939; 2,095,940; and 2,095,941).

DINITROPHENOL AND DERIVATIVES

The high insecticidal values of dinitrophenol and dinitro-orthocresol have been known for some time. In order to lessen their destructive action on plant foliage, which restricts their use, many derivatives have been studied. One of these contains a cyclohexyl radical in the ortho position. This compound is now on the market and has been widely used in California in the form of a dust made by impregnating walnut-shell flour. It has proved effective there for combating the citrus red mite and other insects and, during 1938, 700,000 pounds of it were applied to 7,000 acres of citrus orchards.

The Division of Insecticide Investigations has endeavored to make dinitrophenol and its alkyl derivatives less phytocidal by putting substituents into the hydroxyl radical rather than in the ring. The ethers of dinitrophenol are covered in U. S. patent 2,115,046 and the esters in U. S. patent 2,127,690, both issued to L. E. Smith. It is believed that the compounds described in these patents have insecticidal possibilities equal to

those of the dinitrophenol derivatives now commercially available. In laboratory tests made by entomologists of the Bureau the methyl ether of dinitro-o-cresol was very toxic to mosquito larvae and codling moth larvae and the acetate was toxic to these insects and also to tobacco hornworms.

AZO COMPOUNDS

Several azo compounds, when tested by cooperating entomologists of the Bureau, were found to have insecticidal value, and their use for this purpose was protected by 8 patents issued to Vivian and Haller (U. S. patents 2,094,831; 2,095,939; 2,095,940; 2,095,941; 2,096,414; 2,110,896; 2,110,897; 2,111,879). Some of these azo compounds appear promising for the control of codling moth larvae. Accounts of their toxicity to mosquito larvae have been published by Fink et al. (303, 381).

HETEROCYCLIC COMPOUNDS CONTAINING NITROGEN AND/OR SULFUR

Because of the high insecticidal value of certain compounds containing nitrogen (e.g., hydrogen cyanide, nicotine, anabasine) or sulfur (e.g., carbon bisulfide, mercaptans, and disulfides) or both nitrogen and sulfur (e.g., thiocyanates and isothiocyanates), attention was directed to the preparation of other compounds of these classes. The correctness of this reasoning was proved by the results obtained by Campbell, Sullivan, L. E. Smith, and Haller (233) on testing various synthetic organic compounds against mosquito larvae. Twenty-four out of 68 compounds, mostly sulfur compounds, were found to equal or exceed nicotine in toxicity. For example, diphenylene sulfide killed nearly 100 percent of the larvae in 5 hours at 1:200,000.

A list of organic sulfur compounds used as insecticides (250) was compiled by Roark and Busbey for the benefit of investigators in this field. Many of these compounds contain nitrogen also.

Phenothiazine. -- This heterocyclic compound contains both sulfur and nitrogen and is one of the most promising synthetic organic insecticides that has been developed in the past 5 years. It was reported by Campbell, Sullivan, L. E. Smith, and Haller (233) to be even more toxic than rotenone to mosquito larvae, being very effective at a concentration of 1:1,000,000. Against codling moth larvae in laboratory tests reported by Siegler, Munger, and L. E. Smith (261, 295) it was more toxic than lead arsenate. A review of tests with it against many species of insects was published by L. E. Smith (321). An application by L. E. Smith for a patent on the insecticidal use of phenothiazine is pending in the United States Patent Office.

All derivatives of phenothiazine so far tested have little or no toxicity to insects (Schaffer, Haller, and Fink, 331) or to goldfish (Gersdorff and Claborn, 388). A method of analyzing commercial phenothiazine was described (L. E. Smith, 376), and the ether-insoluble material which it ordinarily contains was shown to be insecticidally worthless (412).

Phenothioxin (phenoxathiin). -- This organic sulfur compound is related to phenothiazine, the NH in the latter being replaced by an oxygen atom. In laboratory tests made by several entomologists of the Bureau, phenothioxin was as effective as rotenone against mosquito larvae in all concentrations up to 1:500,000, and against codling moth larvae it was more

effective than lead arsenate. It was also effective against houseflies when dissolved in kerosene and used as a spray. U. S. patent 2,049,725, issued to L. E. Smith, covers the use as an insecticide of phenothioxin and other diaryl thioxins.

Dimethylacridan. -- This nitrogenous compound is also related to phenothiazine, the sulfur in the latter being replaced by the $(CH_3)_2C$ group. In laboratory tests by Swingle and other entomologists it proved highly toxic to mosquito larvae, cross-striped cabbage worm, cabbage webworm, and cabbage looper. Its insecticidal use is protected by U. S. patent 2,099,826 granted Schaffer and Haller. Dimethylacridan is now manufactured on a large scale for use in the manufacture of rubber goods.

Phenazine. -- This differs from phenothiazine in that the sulfur atom in the latter has been replaced by another NH. Phenazine looks especially promising as a stomach poison to insects, laboratory tests by Siegler with codling moth larvae showing it to be more effective than lead arsenate. Phenazine and related pyrazines are protected in U. S. patent 2,110,614 granted Vivian and Haller.

Xanthone. -- This synthetic is related to phenothioxin, the S in the latter being replaced by the CO group. It has been tested in the laboratory by entomologists of the Bureau and found to be toxic to several species of insects, including mosquito larvae, celery leaf tier, European corn borer, cross-striped cabbage worm, melon worm, southern armyworm, and others. Of especial interest is its effectiveness against codling moth larvae under orchard conditions in the Pacific Northwest. An application for a United States patent covering the use of xanthone as an insecticide has been filed by L. E. Smith

MISCELLANEOUS SYNTHETIC COMPOUNDS

Several hundred organic compounds have been synthesized by the chemists and tested by the entomologists of the Bureau against mosquito larvae, codling moth larvae, or other insects. Reports of these tests have been published by Fink, Siegler, and Munger in cooperation with various chemists (303, 304, 312, 381, 387, 461).

A method of preparing m-bromophenol and m-iodophenol by hydrolysis of the acetate obtained by the action of acetic acid on the corresponding diazonium borofluorides (Haller and Schaffer, 204; L. E. Smith and Haller, 424) was published. This method of replacing the diazo group by the acetoxy group is capable of wide application (L. E. Smith and Haller, 207; Claborn and Haller, 339).

A method of preparing the drug acetphenetidin from p-aminoacetanilide was published by Haller in cooperation with Keenan of the Food and Drug Administration (228); also a report of the action of isobutylmagnesium bromide on 3,4,5-trimethoxybenzonitrile (458).

The commercial position in 1938 of synthetic organic compounds used as insecticides was reviewed by Roark (427). Many synthetics are used as fumigants (e.g., ethylene oxide, methyl bromide, hydrogen cyanide, paradichlorobenzene), but only a few as contact insecticides (thiocyanates, iso-

thiocyanates, derivatives of dinitrophenol and of cyclohexylamine), as stomach insecticides (phenothiazine), or as repellents (tetramethyl thiruam disulfide). The history and principal uses of these compounds were discussed briefly.

RELATIONSHIP BETWEEN TOXICITY AND CHEMICAL CONSTITUTION

If the relationship between the chemical constitution and physical properties of organic compounds and their toxicity to insects were understood, the synthesis of potent insecticides would be a comparatively simple matter. Unfortunately, organic compounds are highly specific in their action on insects, and a material effective against one species may be ineffective against others. Rotenone, for example, which is very toxic to the imported cabbage worm, Pieris rapae L., can be eaten with impunity by several species of borers. There is no way of predicting such difference in behavior, and one cannot find an insecticide suitable for codling moth control by means of tests upon aphids or houseflies, or even upon other kinds of lepidopterous larvae.

When different materials are tested upon the same stage of the same species of insect under identical conditions, certain relationships between toxicity and physical and chemical characteristics are discovered, however, and the Division of Insecticide Investigations, in cooperation with many entomologists of the Bureau, has taken an active part in the search for such relationships and has been successful to some degree. Several instances are given in the section on synthetic organic insecticides.

The problem appeared so important the Division instituted a fundamental investigation of the toxicity of organic compounds in which, because of their ease of handling throughout the year, cheapness, and susceptibility to poisons, goldfish are used as the test animals. A method based on observation of survival time was developed (70) and tested throughly in connection with a study of the photochemical decomposition of rotenone and related materials (177); very definite relationships were shown among the hydro, hydroxy, and acetate derivatives of rotenone (258); all commonly accepted methods of calculating comparative toxicities of substances were considered (86, 103, 174, 189, 213, 258, 270, 323, 335, 415), and a new criterion of toxicity was developed (257) and used in subsequent work. Realizing that a fundamental study such as this should begin with the simpler compounds, a long-time study of phenol and its derivatives was begun. It has been demonstrated that the replacement of the oxygen of the phenolic nucleus with sulfur greatly increases toxicity (323, 415); the introduction of the nitro group (460), the chlorine atom, and the bromine atom into phenol showed that the para derivative is in all cases the most toxic, and is appreciably more toxic than phenol. The fish tests have also been used to demonstrate the superiority of optically active compounds over optically inactive ones, as exemplified by the dihydrodequelins (270); and also have been employed in the comparison of nicotine and anabasine (189) and in the study of the relationships between phenothiazine and its oxidation products (383). All the tests with goldfish have been conducted by W. A. Gersdorff, and most of the publications cited are his.

In cooperative studies to ascertain the relationship between chemical constitution and toxicity to codling moth larvae a number of halogen and nitro compounds of benzene were investigated by Siegler, Munger, and L. E.

Smith (435). Neither fluoro-, chloro-, bromo-, iodo-, or nitrobenzene, nor the fluoro- or chloronitrobenzenes (ortho, meta, and para) were toxic. Of the bromo- and iodonitrobenzenes the para derivative was the most toxic. None of the dihalogenated (dichloro, dibromo, and diiodo) benzenes was toxic, and of the dinitrobenzenes the meta isomer is much more toxic than the other two. It was concluded that the data obtained indicate no marked correlation between either the groupings involved or their relative position with regard to their toxicity to the codling moth larvae.

INORGANIC INSECTICIDES

At present the control of injurious insects is largely accomplished by the application of inorganic insecticides, and this practice will continue until suitable organic materials can be developed. Because organic insecticides are specific in their action, whereas inorganic insecticides such as the arsenicals are effective against a wide range of pests, it will be a long time before the latter are displaced, and so long as they are used, any information that will lead to a better knowledge of how to use them is of benefit. The Division of Insecticide Investigations has made important additions to the fund of information concerning many of the inorganic insecticides, which will be briefly described in the sections that follow.

The arsenicals continue to be the most important group of inorganic insecticides, and several of these have been investigated.

LEAD ARSENATE

More than 40 million pounds of this arsenical are used annually in the United States. The principal advances made in our knowledge of lead arsenate relate to its distribution on sprayed fruit and its removal therefrom, and are the result of cooperative studies by chemists, entomologists, and horticulturists. These aspects are discussed in the section on spray residues. Certain compatibility studies have been made, however, by Murray concerning its use with oil emulsions (141) and spreaders (154). The relative efficiency of various spray schedules using lead arsenate has also been investigated by Steiner, Sazama, Fahey, and Rusk (301). Recently a survey has been made of all the lead arsenates on the market with respect to particle size. A study of the question of the use of lead arsenate in soils to control the Japanese beetle grub led to the discovery by Mell that greensand marl and limonite both have practical value in counteracting the phytocidal action of the arsenic.

CALCIUM ARSENATE

Commercial calcium arsenate, of which over 40 million pounds are used annually for insecticidal purposes, is not a simple chemical but a complex and variable mixture. Using a method for determining free lime in it, which was developed in 1924 by C. M. Smith (Ind. & Eng. Chem. 16: 950. 1924), the Division has made two surveys (Smith and Murray, 91; Nelson and Cassil, 344) of all the products on the American market and demonstrated that very great chemical differences exist between materials which, by the ordinary methods of control analysis, appear identical. A survey of the physical characteristics of commercial calcium arsenates was made by Goodhue (343) which gave quantitative data for the first time concerning the large differ-

ences in particle size which exist among them. In the course of this work an actual chemical separation was accomplished by means of a mechanical classifier (Goodhue and Cassil, 336), confirming the complexity mentioned above. A phase-rule study of the calcium arsenate system by Nelson of this Bureau and Haring of the University of Maryland (363) added to our information on this important insecticide. A new chloroarsenate of calcium (268) was prepared by C. M. Smith. A new process for preparing calcium arsenate was developed by means of which the composition can be more definitely controlled and the solubility kept low (Nelson, 452).

The effects of heat treatments of some calcium arsenates on their toxicity to silkworms and bean plants was reported on by Bulger and Nelson (474) in 1939.

Four hydrous calcium arsenates of typically different composition were ignited to produce the corresponding anhydrous preparations, and eight portions of a sample of commercial tetracalcium arsenate were autoclaved or treated with dry heat. A portion of each preparation was then fed to silkworm larvae, Bombyx mori L., by the sandwich method, and their toxicity determined in terms of the median lethal dosage range. Plant tolerance of these calcium arsenate preparations was determined by applying them to bean foliage in the greenhouse and estimating the degree of foliage destruction.

All the hydrous calcium arsenates were fairly toxic to silkworm larvae. Monocalcium and dicalcium arsenates were the most toxic and about equal in toxicity, notwithstanding the fact that the amounts of soluble arsenic present were greatly different. Tricalcium and tetracalcium arsenates were only about half as toxic as the monocalcium and dicalcium compounds. The anhydrous form of each compound tested was nontoxic to the extent that when large doses were fed (12 to 50 times the M. L. D. for hydrous forms) no median lethal dosage range was established.

In general, ignition decreased the percentage of soluble arsenic present. However, there does not appear to be any appreciable correlation between the percentage of soluble arsenic as determined by the Association of Official Agricultural Chemists method and the toxicity to insects or plants.

Heat treatments of calcium arsenate sufficiently severe to make the product relatively safe to bean foliage likewise make it nontoxic to silkworm larvae. The fact that autoclaving at 100° C. for 1 hour does not destroy the toxicity, whereas the same treatment for 3 hours produces a relatively nontoxic tetracalcium arsenate, suggests that the toxicity balance of such a calcium arsenate is rather delicate and indicates that great care should be exercised in its preparation if the toxicity is to be maintained.

Dry heating at controlled temperatures does not affect the toxicity of tricalcium arsenate so readily as that of tetracalcium arsenate.

PARIS GREEN AND HOMOLOGS

Paris green is used to the extent of about 3,000,000 pounds annually. In 1937 the Division reported a survey of all the greens on the American market, both as to chemical composition (Dearborn, 357) and particle size distribution (Goodhue and Gooden, 368). The latter constituted the first

available information of this kind. Dearborn also discovered that other acids than acetic acid were capable of forming complex compounds with copper arsenite similar to paris green, and methods were developed for producing such homologs from the lower and higher members of the acetic acid series (260, 285), from the members of the oleic and linoleic acid series (324), and from the acids contained in various other animal and vegetable oils (369). Some of these greens have insecticidal power superior to that of paris green and at the same time are less injurious to plants. Three patents covering them have been granted Dearborn (U. S. patents 2,080,004; 2,104,584; 2,127,380).

Reeves of the Bureau of Plant Industry and Yothers and Murray of this Bureau (466) in 1939 reported that when mixed with vegetable oils and applied as wound dressings to apple trees to prevent the aphid infestation that is responsible for the growth of perennial canker, the stearo-, palmito-, and lauro-arsenites of copper permitted the fungus to advance into tissue containing a total average of 256 parts per million As_2O_3.

OTHER ARSENICALS

Dearborn examined all the brands of magnesium arsenate (71) and of manganese arsenate (63) on the American market, and compiled the information, both chemical and insecticidal, which has been published concerning these two insecticides (411 and 337, respectively). Magnesium arsenate was at one time of particular importance as the only recognized means of controlling the Mexican bean beetle, and the proprietary insecticide Manganar, promoted as a substitute for lead arsenate, contains manganese arsenate.

FLUORIDES

The Division was among the first to recognize the possibility of finding acceptable horticultural insecticides among the compounds of fluorine, one member having been granted a patent on the subject in 1925 (Roark, U. S. patent 1,524,884). The possibilities of almost all available fluorine compounds were surveyed, and their insecticidal properties called to the attention of the public (Carter, 55). Newcomer and Carter (201) reported studies of fluorine compounds for controlling the codling moth. Special attention was given to the fluoaluminates (68), of which cryolite is one. The solubilities of numerous fluorides and fluosilicates were determined for the first time by Carter (22, 67); and the relative toxicity as stomach poisons to insects of some of them was measured by Shepard and Carter (195). The compatibility of fluosilicates and of cryolite with arsenical compounds was demonstrated (38), and the incompatibility of all fluosilicates with lime (97) and of barium fluosilicate with nicotine sulfate (157) was pointed out, also by Carter. Two surveys of the fluorine compounds on the American market were made (Carter and Roark, 21; Carter, 156), and the attention of entomologists was drawn by Carter to the fact that the product commonly called by them calcium fluosilicate is not actually that (146). A review giving all available references to the insecticidal uses of fluorine compounds was prepared by Carter and Busbey (311, 432). Three patents for the preparation of insecticidal compounds of fluorine have been granted Carter (U. S. patents 1,842,443; 1,863,266; 1,863,519).

Because cryolite has become the most important of the fluorine com-

pounds used as agricultural insecticides, a special study was made in 1939 of all cryolite materials offered for insecticidal use. The fluorine content of 18 samples of cryolite, comprising natural, synthetic (domestic and imported), and diluted materials, was determined, and the equivalent content of sodium fluoaluminate calculated. Samples of domestic synthetic origin showed an average of 82.8 percent sodium fluoaluminate, samples of foreign synthetic origin 91.3 percent, and samples of natural origin 88.2 percent. Dusting materials showed different sodium fluoaluminate content, depending on the amount of diluent added.

Solubility determinations were made on all samples by two methods, (1) chemical analysis and (2) evaporation and weighing of the residues. Results by the two methods were in agreement. The solubilities of the three classes, synthetic domestic, synthetic imported, and natural, were very nearly the same and in close agreement with the solubility figure for cryolite as reported in chemical handbooks (Carter, 463).

MERCURIC CHLORIDE

This compound has been used for the control of gladiolus insects, in the form of a dilute solution as a dipping bath for the corms. Cassil, in cooperation with R. H. Nelson of the Division of Truck Crop and Garden Insect Investigations, demonstrated for the first time that the corms adsorb the chemical from solution and thereby reduce the concentration to about half of that necessary for control, which is of great importance, considering the practice of using the same solution for several lots of corms.

COPPER COMPOUNDS

The compounds of copper are primarily fungicides, but have certain insecticidal properties as well. Our work has resulted in a better understanding of the basic sulfates of copper (Nelson, 14), which are the principal constituents of bordeaux mixture, and cooperative experiments with Leukel of the Bureau of Plant Industry showed that among all the copper compounds studied, the basic sulfate, the basic carbonate, and compounds of aniline with copper sulfate and with copper chloride were most serviceable in controlling stinking smut of wheat (364). The copper-aniline compounds were given their first trial as fungicides in these experiments.

Commercial copper fungicides, consisting essentially of copper phosphate, copper oxychloride, copper zeolite, basic copper sulfate, and a copper-zinc compound, were found to affect adversely the toxicity of the tank-mix nicotine bentonite-soybean oil used successfully in Indiana for codling moth control. These copper compounds also produced very serious foliage injury and reduced the resistance of the nicotine deposits to the weathering effects of heavy rains (Steiner and Fahey, 451). There is great need of fungicides that are compatible with nicotine bentonite.

SULFUR

Because of the great effect that particle size has on the efficacy of dusting and spraying sulfurs, a survey of 44 products typical of those on the American market was made, and for the first time a clear picture was given to entomologists of the great range of average particle size

(over 10 times) which may be encountered in insecticidal sulfurs (Goodhue, 396).

SPRAY RESIDUES

The Division of Insecticide Investigations has been prominently identified with the spray-residue problem almost from its beginning in 1925. In that year England placed an embargo on all apples from the United States that contained more than 0.01 grain of arsenious oxide per pound. Control of the codling moth in the Pacific Northwest required the use of so much lead arsenate spray that the residue at harvest invariably amounted to more than this, and in order to save the export business in apples (amounting to 7,098,000 barrels for the year ending June 1927) it became necessary to study means of keeping the residues down and of removing those which could not be avoided. In 1928 the Division established a field station at Yakima, Wash., in conjunction with the Bureau's fruit insect field station there. Later that year the chemical work was transferred to Wenatchee, Wash., and in 1935 it was again placed in Yakima. Work was done with cooperating entomologists to improve spraying schedules, and with cooperating horticulturists of the Bureau of Plant Industry to develop fruit-washing procedures effective in meeting the arsenic tolerance established by the Department of Agriculture. When in 1933 the Department established a tolerance for lead, it necessitated a renewed study of lead arsenate residues, and the establishment of a tolerance for fluorine in 1934 required an extension of our work to include that element.

Every year thousands of analyses of washed and unwashed samples of apples have been made both at Yakima and at Washington, D. C., to determine the efficacy of various washing solutions in removing residues of lead arsenate. The results of these tests, which were carried on cooperatively with Diehl, Ryall, Smith, and Haller of the Bureau of Plant Industry, Beaumont of the University of Maryland, Gould of the West Virginia Agricultural Experiment Station, and Newcomer of this Bureau, have been published in a series of articles (32, 39, 72, 81, 183, 186, 212, 225, 242, 278, 398), and two patents covering processes of washing arsenical residues from apples have been obtained (Robinson, U. S. patents 1,884,966 and 1,885,100). At the same time, the influence of fish oil, mineral oil, and other supplementary materials upon the magnitude of the residues at harvest were studied and described in cooperation with Diehl, Ryall, and Haller of the Bureau of Plant Industry and Beaumont of the University of Maryland (142, 153, 183, 252).

The effect of apple growth and of weathering in altering deposits of lead arsenate throughout the growing season was determined by Carter and Newcomer (186).

Experiments were undertaken in southern Indiana by Fahey in 1935 and 1936, in cooperation with Steiner and associates of the Division of Fruit Insect Investigations, to determine the extent of decomposition of lead arsenate spray residues on apples as measured by their ratios of lead to arsenious oxide. The residues were analyzed immediately after the various cover-spray applications and again after the residues had weathered. A total of 248 samples of apples and apple foliage were used. The average ratio of lead to arsenious oxide in these samples did not vary significantly from that in the original spray material, and samples taken immediately after spraying showed no significant difference from those taken after weathering. The slight variations between the ratios are in reality the

combined analytical errors of the methods of residue analysis. The high ratios obtained by early investigators are due, probably, to inadequate samples or to unreliable methods of analysis (439).

In cooperation with Heller of the Bureau of Plant Industry and Gould of the West Virginia Agricultural Experiment Station, the variability of residues of lead arsenate within individual spray treatments was investigated (40, 325, 359) in order to determine the procedure necessary to obtain adequate samples in all investigational work. As the result of cooperative work with Wichmann and associates of the Food and Drug Administration, improvements have been made in the analytical procedure for lead (208). Methods for the determination of arsenic (48, 49, 168, 196, also unpublished recent work) have also been improved.

Parallel with the work on residues of lead arsenate, similar studies have been carried out by Carter on fluoride residues, mostly residues of cryolite on apples (158, 226, 267), and three patents covering new washing solutions especially adapted for cryolite removal were granted to him (U. S. patents 2,046,546; 2,046,547; 2,046,548).

The Division has continually kept in mind the necessity of studying residues of other materials for which no tolerances have been established. In this work the methods to be used in determining the residues have had to be developed, as described in the section on analytical investigations. The influence of the characteristics of oil emulsions upon the amounts of oil deposited was shown by studying the oil residues (Cressman and Dawsey, 224); the removal of nicotine bentonite residues from apples has been shown to be easily accomplished (Fahey, Rusk, Steiner, and Sazama, 407); and the presence of lead and arsenic in various forms of tobacco, and of arsenic in the smoke therefrom, has been studied (205, 305). An exhaustive study (awaiting publication) of the derris residues on cabbage has been made, giving the only data available on this important subject. The entomologists of the Bureau have been given the results of our determinations of residues of phenothiazine on cherries, sulfur on citrus, tartar emetic on citrus, borax on rice, etc., all of which are of great importance to them in shaping their spraying and dusting programs. A study by Murray and Ryall (454), reported in June 1939, showed that phenothiazine spray residues on apples in amounts up to 0.060 grain per pound may be readily reduced to 0.01 grain per pound or less with present washing equipment and solutions (e.g., 1.5 percent HCl at 110° F. for 30 seconds).

A method was described for spraying paraffin-coated mica plates with oil emulsions and determining the quantities of oil deposited by difference in weight (Dawsey, Cressman, and Hiley, 330). Comparison between the quantities of oil deposited on the paraffin surfaces of the plates and those deposited on chrysanthemum foliage, as determined by chemical analysis, showed that various emulsions give different ratios of plant deposit to plate deposit. It is, therefore, not practical to attempt to standardize the sprays intended for the plant surface against the plate surface.

The development by Dawsey and coworkers of analytical methods for accurately determining residues of petroleum oils on leaves (161, 292, 371) enabled studies to be made of the relation between oil deposit and insecticidal efficacy (Cressman and Dawsey, 299). The comparative insecticidal values of three petroleum oils with 6, 16, and 33 percent by volume, re-

spectively, of sulfonatable material were investigated in laboratory and field spraying tests. The oils were applied, in the form of emulsions containing from 0.9 to 2 percent of oil, sodium oleate soap being used as the emulsifier, to camphor-tree plants and trees infested with the camphor scale. The other characteristics of the different oils were substantially the same, so any detectable differences in scale mortality could be due only to differences in the sulfonatable portions. When sprays which gave equal oil deposits were compared, the variations in mortality appeared to be entirely random, and no differences were found which could be attributed to the sulfonatable content of the oils. Analyses of the oil deposits left by sprays showed that substantially equivalent deposits were obtained when equal concentrations of the different oils were applied. In the laboratory tests the oil deposits produced were independent of the sulfonatable content of the oil but varied directly with the oil content of the spray.

The chemical methods of studying codling moth insecticides at Vincennes, Ind., were described in 1939 under the title "A field method for the chemical evaluation of spray deposits resulting from the application of insecticides for control of the codling moth" by Fahey and Rusk (471). The methods of sampling and chemical analysis are described for residues of lead, arsenic, nicotine, and phenothiazine. The weathering of residue deposits and the relationship of residues to area of fruit are discussed.

Fahey, Rusk, Steiner, and Sazama (475) in 1939 reported on the ease of residue removal from late and early spray applications of lead arsenate to apples. Spray schedules designed to permit such a comparison were applied in experimental apple orchards near Vincennes, Ind. Samples of fruit from the sprayed plots were washed with hydrochloric acid solutions, and analyses were made for lead residues before and after washing.

The residue load at harvest from a given number of lead arsenate sprays increases as the time of spray application approaches harvest time; thus, a final cover spray of lead arsenate may leave, at harvest, a residue load equal to that resulting from four first-brood sprays. The residues from sprays applied in the second-brood period are more easily removed by washing than are residues from the same number of the same sprays applied during the first-brood period. The use of oil adhesives with organic insecticides, in second-brood sprays, makes the residue of lead arsenate from first-brood sprays as difficult to remove as are residues from lead arsenate used throughout the season.

A digest of the literature through 1934 relating to insecticidal spray residues (431) was issued by Busbey early in 1939.

FUMIGANTS

From its creation in 1927, the Division of Insecticide Investigations has been active in the study of fumigants. In cooperation with entomologists, it tested several hundred organic compounds (Roark and Cotton, 54) in an endeavor to find substitutes for the dangerously inflammable and explosive carbon disulfide. As a result several new fumigants now in commercial use were discovered, such as methyl and ethyl formates (8), ethylene dichloride (2), propylene dichloride (6), and ethylene oxide (16). These are discussed below in detail. The high insecticidal value of the vapors of other compounds was also discovered, for example, those of chloroacetates

(U. S. patent 1,789,322) and other halogenated fatty esters (11). These discoveries were all made jointly with R. T. Cotton.

The use of carbon dioxide to increase the insecticidal efficacy of fumigants was discovered by Cotton and Young (30), and mixtures of carbon dioxide with ethylene oxide and with methyl formate have been marketed for use as fumigants. Back, Cotton, Young and Cox (64) described the use of the ethylene oxide-carbon dioxide mixture for treating stored grain.

In cooperation with the Division of Fruit Insect Investigations many organic compounds were tested for toxicity upon the California red scale (Cupples, Yust, and Hiley, 298) in an effort to find substitutes for hydrogen cyanide. Methyl thiocyanate (Lathrop, Cupples, Hiley, and Yust, 284) proved toxic to the scale but was also injurious to citrus trees. The "interval shooting" or piecemeal application of hydrogen cyanide in citrus orchards was discussed (Cupples, 172), and the latest developments in the fumigation of mushroom houses with sulfur dioxide (Davis and Young, 216, 246) and with hydrogen cyanide (Davis and Claborn, 272) were described.

The fumigation of cereal products, flour, and flour mills with hydrogen cyanide and other fumigants was described (Cotton, Wagner, and Young, 273, 294, 309, 336, 361); deficiency of oxygen as a factor in killing insects in vacuum fumigation was discussed (346); and the use of heavier-than-air fumigants on insect-infested grain and other products was reported on (358). These studies were made by Cotton, Wagner, and Young at Manhattan, Kans. The rate of evolution of hydrogen cyanide from calcium cyanide was studied (37). The effect of a number of fumigants on the germination of seeds was investigated by Young and Roark (45, 54).

Many studies of the vapor pressures of fumigants were made by Nelson and Young (23, 24, 27, 73, 185), and improved apparatus for measuring partial vapor pressures of binary liquid systems was perfected by Nelson (135). From these data were calculated the maximum weights of various fumigants which can exist in vapor form in a 1,000-cubic-foot fumigating chamber (29), which were published by Nelson and Roark, who also compiled data on the densities of mixtures of air and various fumigants (84).

Methods were devised for determining small quantities of methyl bromide in air (Busbey and Drake, 400), and for determining hydrogen cyanide in a mixture of air and carbon dioxide (Cupples, 167). Apparatus for the rapid vaporization of carbon disulfide (Weigel, Young, and Swenson, 4), for burning sulfur for mushroom house fumigation (Davis and Young, 237), for laboratory fumigations with hydrogen cyanide under controlled temperature and humidity (Cupples, 166), and for fumigating aphids under laboratory conditions with minute quantities of nicotine (H. H. Richardson and Busbey, 350) was devised and described. The binary system carbon tetrachloride-ethylene dichloride which is used as a fumigant (2) was studied, and the boiling points and specific gravities were recorded as aids in analysis (Young and Nelson, 128).

The work of investigators of fumigants has been aided by the publication of bibliographies of some of the more important materials, for example, those on chloropicrin by Roark, Gersdorff, and Busbey (79, 209, 253), that on ethylene dichloride by Gersdorff (132), that on ethylene oxide by Young and Busbey (247), and those on cyanides by Cupples, Young, and Busbey

(259, 281, 288, 470, 476). Popular articles and reviews of progress in fumigants have also been published by Roark (5, 10, 57, 105, 116, 140, 143, 218, and 293).

The volume of popcorn was found to be affected by certain fumigants used to treat the corn when newly harvested for the destruction of insects. Fumigating the corn with hydrocyanic acid improved its popping quality, carbon disulfide and ethylene oxide at regular dosages had little effect, whereas carbon tetrachloride, ethylene chloride, and chloropicrin had adverse effects. In all cases at the end of 30 days the deleterious effects had completely disappeared and the volume of the popped samples was again at normal (Young, Cotton and Wagner, 428).

FORMATES

The high insecticidal value of the volatile esters of formic acid was discovered by this Division working in cooperation with the entomologist R. T. Cotton (8, 54). Methyl formate and ethyl formate have come into commercial use for the fumigation of raisins, dried fruits, nuts, and other foodstuffs and also stored tobacco and other commodities. A mixture of methyl formate and carbon dioxide has been used commercially also. The vapor pressures of the formates were determined (24), and from these data were calculated the maximum weights of the various formates that can exist in vapor form in a 1,000-cubic-foot fumigating chamber (29). This information is helpful to the practical fumigator.

ETHYLENE DICHLORIDE AND PROPYLENE DICHLORIDE

The value of these two organic solvents as insecticidal fumigants was reported (Cotton and Roark, 2, 6, 54). In order to reduce the slight fire hazard of ethylene dichloride, it is mixed with one-third its volume of carbon tetrachloride (2). Both ethylene dichloride and propylene dichloride are now widely used for killing weevils in grain and clothes moths in furs and woolen clothing. These fumigants are now used in place of cold storage by many warehouses storing furs, clothing, and household goods because their use is cheaper and more effective in killing insects. They are inexpensive (costing about 5 or 6 cents per pound), readily available, relatively nontoxic to man, and leave no objectionable odor in fumigated articles. A physico-chemical study of mixtures of ethylene dichloride and carbon tetrachloride was made and their boiling points and specific gravities were recorded as aids in analysis (128). A comprehensive bibliography of ethylene dichloride complete through 1930 was published (132).

ETHYLENE OXIDE

This fumigant is now widely used for destroying insect life in all kinds of stored products, especially packaged cereals, bagged rice, tobacco, clothing, and furs. It is best employed in vacuum fumigation chambers. Ethylene oxide is also used for destroying insects infesting books and papers. The Federal Archives Building in Washington, D. C., in which are stored priceless historical documents, is equipped with vacuum fumigation apparatus in which ethylene oxide is used to treat insect-infested material. A recent use of ethylene oxide is to destroy molds, fungi, and other plant life infesting spices, especially packaged spices. Ethylene oxide is the material par excellence for this purpose because it is highly effective at

an economical dosage and leaves no odor, flavor, or deleterious residue in the fumigated product. The use of ethylene oxide for combating economically injurious fungi as well as insects is covered by U. S. patent 1,791,429 granted Roark and Cotton. The use as a fumigant of the mixture of ethylene oxide with carbon dioxide is also protected (Cotton and Young, U. S. patent 2,024,027). These inventions are the result of cooperative research by chemists and entomologists and are more fully described in reports by Cotton and Roark (16, 54) and by Back et al. (64). A bibliography on the use of ethylene oxide for pest control was published (247).

CHLOROPICRIN

This compound, which was extensively used as a tear gas in the World War, has valuable insecticidal, fungicidal, and bactericidal properties. It is free from the fire and explosion hazards of carbon disulfide and other fumigants, and, because of this and other desirable properties, it has come into extensive commercial use for the fumigation of grain, ships' holds, and warehouses, and injurious insects, nematodes, and micro-organisms in soil. We have directly stimulated the use of chloropicrin by issuing three bibliographies (79, 209, 253) on the subject. These contain abstracts of many publications unavailable to most scientific workers, such as works written in Russian.

HYDROGEN CYANIDE

This fumigant continues to be the most important of all. In spite of a history of use of 50 years, there are many problems connected with its application against specific pests that require study. At Whittier, Calif., the Division of Insecticide Investigations, in cooperation with entomologists of the Division of Fruit Insect Investigations, has carried on chemical investigations since 1930 on the fumigation of the California red scale with hydrogen cyanide.

Experimental field fumigation investigations added to our knowledge of the concentrations of hydrogen cyanide in tents during regular commercial treatments and provided dosage data for comparison with insect mortalities in seasonal fumigation studies. The multiplicity of variables in field experimentation, many of which were not amenable to control, coupled with the impossibility of reproducing results, made the identification and evaluation of the factors responsible for wide variation in the insect mortality practically impossible, and ultimately led to the postponement of field work of this nature in favor of laboratory investigations. However, the study of field fumigation yielded data from which a mathematical formula was developed that expresses the drop in hydrogen cyanide concentration with time under a fumigation tent. From a practical standpoint the results show that the use of a more nearly gas-tight tent and thorough mixing of hydrogen cyanide with the air under the tent will assure better insect kill.

For the laboratory studies of hydrogen cyanide fumigation a special apparatus was devised and constructed. By means of this it is possible to control temperature, humidity, and hydrogen cyanide concentration and to expose the insects to the fumigant at once, thus avoiding the effects of protective stupefaction. Reports of these studies are being prepared for publication.

CHLORINE

The control of stinking smut of wheat, oats, barley, and sorghums by the application of gaseous chlorine was investigated by Leukel (473) of the Bureau of Plant Industry and Nelson of this Bureau. Chlorine gas is capable of killing smut spores of grains under carefully controlled conditions but offers greater promise in controlling fungi on garden seeds.

ACCESSORY MATERIALS

It is comparatively seldom that an insecticidal material is used alone. Dusting materials are commonly mixed with inert powders to aid in their distribution, and spraying materials frequently have added to them wetting and adhesive agents to improve their application and adherence to fruit and foliage. A clear understanding of the roles played by these supplementary materials is essential to the proper conduct of chemical control of insects, and the Division has, therefore, devoted considerable attention to them and has made worthwhile advances in our knowledge of them.

WETTERS AND SPREADERS

Cupples has developed a quick and easy method of determining the spreading power of solutions over a standard oily surface (266), and by its means has made a detailed study of the spreading ability of sodium oleate (266, 275), of potassium and ammonium oleates (283), and of the sodium salts of numerous other fatty acids (349, 472), as well as of triethanolamine oleate (379). A survey of the chemical nature of certain spreaders used with lead arsenate was made (154), and a comprehensive list of the more modern wetting agents, including measurements of their spreading coefficients, was published by him (383). The relation of wetting power to ability of liquids to penetrate wood and the relation of initial spray retention to spreading power have been studied. Among the solid, powdered supplementary materials, bentonite has been given special attention, and the role which it plays in the formation of nicotine bentonite and other salts with organic bases and alkaloids was explained for the first time by C. R. Smith (222).

In a study by Cupples (468) of inorganic salts as adjuvants for increasing the wetting power of a proprietary wetting agent (a sulfonated ester of a dicarboxylic acid) it was found that no precipitation occurred in the presence of large amounts of calcium or magnesium. Additions of chlorides of calcium, magnesium, or sodium to solutions of this wetting agent produced significant increases in wetting powers, as measured by surface tension or by spreading coefficient on mineral oil. The improvement is so substantial as to indicate that this effect may be of practical importance in the formulation and use of these materials.

An investigation by Cupples (472) of the wetting and spreading properties of aqueous solutions containing mixtures of sodium carbonate with n-caproic, n-caprylic, n-capric, lauric, myristic, and palmitic acids led to the conclusion that for aqueous mixtures of sodium carbonate with a series of fatty acids at a concentration of 1.0 percent of fatty acid, the surface tension, interfacial tension against mineral oil, and spreading coefficient, when plotted as functions of the alkali-fatty acid mole ratio, give curves which are similar in form with few exceptions. The relative

positions of the curves correspond approximately with the order of increasing molecular weight of the fatty acids.

The characteristics of the carbonate mixture are similar to those of the corresponding hydroxide mixtures, with some differences which are apparently accounted for by the diacidic nature of the carbonate. The oleate mixtures have exceptional properties which help to explain their excellence as detergents.

DILUENTS

A comprehensive study of the physical characteristics of diluents suitable for use in making dusting mixtures is under way at present.

STICKERS

The effects of the use of fish oil and mineral oil as adhesives with lead arsenate upon the magnitude of the deposits obtained was investigated by us in cooperation with Diehl (142) and Ryall (153) of the Bureau of Plant Industry.

A new adhesive especially adapted for causing derris powder and similar organic materials to stick to foliage was invented by Goodhue (U. S. patent 2,129,517). This consists of rosin residue or pine oil foots, a substance that remains in the fractionating column after the removal of solvent and pine oil in the steam and solvent process for the production of naval store products from pine wood. This adhesive is best handled when emulsified in water with ammonium caseinate. It is noninjurious to foliage, does not accelerate the decomposition of rotenone and related compounds, and retains 85 to 95 percent of the derris on bean foliage after a washing test equal to 2-1/2 inches of rain in a period of 5 minutes. The concentrated emulsion is commercially available and is widely used, especially for combating the Japanese beetle with derris.

Additional stickers that have been developed by us are the reaction product of furfural with aniline (Goodhue, U. S. patent 2,146,257) and the reaction product of furfural with acetone (Goodhue, U. S. patent 2,146,258).

EMULSIFIERS

A formula for the preparation of insecticidal oil emulsions using casein ammonia as the emulsifying agent was developed by Newcomer and Carter (194). Emulsions made in this way have been thoroughly tested in the laboratory and in the orchard, and shown to be practical, economical, and effective, and fruit growers in the Pacific Northwest are using them successfully on a large scale. The influence of various emulsifiers on the quantity of oil deposited from emulsions was determined by Cressman and Dawsey (224).

ANALYTICAL INVESTIGATIONS

The Division of Insecticide Investigations, in the course of its developmental work, is constantly concerned with the improvement of existing methods of analysis and the development of new methods when none are available. This has led to some notable contributions that have been of

great use to every one interested in insecticides.

Because of the importance of arsenic as an economic insect poison, analytical methods for its determination and for the examination of insecticides and foodstuffs containing it are extremely important. The Division has always taken a leading part in this work, some one of its members having acted for years as the Referee on Arsenic for the Association of Official Agricultural Chemists, conducting the collaborative work for the Association and submitting regular annual reports (251, 338, 390, 444). Numerous important improvements have been made by us in the Gutzeit method of determining the micro-amounts of arsenic that are encountered in spray-residue work. Improved methods for the preliminary digestion of the samples have been worked out by Barnes (49), the accuracy of the method has been determined by him and Murray (48), and means of overcoming certain interferences have been worked out by Gross and Cassil (168, 196, 338). Recently, in cooperation with Wichmann of the Food and Drug Administration, a new method (446) for micro-amounts of arsenic has been devised by Cassil, which is equally as speedy as the Gutzeit method and far more precise. A procedure was published for determining arsenic in soil treated with acid lead arsenate (Koblitsky, 462).

The Division has also developed a method for free lime in calcium arsenate that has been the most important factor in unravelling the make-up of this most complex insecticide (91). Recently, tartar emetic has assumed some importance as an insecticide, and because of the lack of a suitable method for the determination of antimony in spray residues from its use, a new method was developed in cooperation with Davidson and Pulley of the Bureau of Agricultural Chemistry and Engineering (391). Advances have been made in the analysis of foodstuffs for lead (Wichmann et al., 208), and a specific method was worked out for lead in chewing tobacco and snuff (Cassil and C. M. Smith, 305). A method was developed for residues of barium fluosilicate (Carter, 102) and other fluorine compounds (Carter, 354), and for the copper, arsenic, zinc, mercury, and fluorine (Carter, Capen and Cassil, 403) left in wood that has been impregnated with their soluble salts.

The study of natural insecticides of plant origin has resulted in the development and publication of methods for nearly all the important compounds of this class. The Division has been the pioneer in respect to methods of examining rotenone-bearing plants. The discovery by Jones that rotenone forms sparingly soluble complexes with carbon tetrachloride and dichloroacetic acid led to the development of a macro method (122, 165, 334, 423) for the estimation of rotenone in roots. In cooperation with Graham (373, 377) of the Food and Drug Administration, who also is Referee on Insecticides for the Association of Official Agricultural Chemists, the method was further improved and has been practically universally adopted; the Food and Drug Administration now uses it in control work, and the Association of Official Agricultural Chemists has taken action to make it an official method. One qualitative color test was developed (Jones and C. M. Smith, 169) which permits a rapid testing in the field during search for rotenone-bearing plants, and such use has actually been made of it in the researches of the Department as well as elsewhere. Another color test for micro amounts of rotenone and deguelin was devised by Gross, Smith, and Goodhue (219, 276) which permits of quantitative application, and this test has been used by numerous investigators all over the world as a quick means of studying the decomposition of derris and the isolation of constituents

from it. Recently a comparison has been made of all published color tests. The possibility that optical rotation of the extracts might serve for testing rotenone-bearing plants was looked into by Jones (322). In cooperation with Sullivan, chemical means of estimating the insecticidal efficacy of derris or cube (289, 395) were developed by Jones and Smith which are definitely of service in comparing various samples.

The methods now used for the determination of the two active principles in pyrethrum powder are not wholly satisfactory. We have contributed a new procedure for estimating pyrethrin II (Haller and Acree, 263) and at present have under study another entirely new procedure for estimating both pyrethrins I and II separately.

Our contributions to the analysis of nicotine products have been particularly notable. One micro method, suitable for fumigation concentrations, based on weighing the silicotungstate compound (Spies, 317, 320), and another based on the photoelectric estimation of the same precipitate (Goodhue, 374), were worked out. Two methods specifically adapted for determining nicotine in spray residues were developed (Markwood, 378, 445), the latter colorimetric procedure permitting the determination of the minute quantity of nicotine spray residue on one apple.

The attempt to develop synthetic organic insecticides has also led to numerous important advances in analytical technic. The examination of one of the fumigants developed by the Bureau, namely, the mixture of ethylene dichloride and carbon tetrachloride, was facilitated by the determination of the boiling points and specific gravities of all mixtures of the two components (Young and Nelson, 128). The study of fumigation problems was aided by a new method of determining hydrocyanic acid (Cupples, 167); a method for the macro-examination of phenothiazine was developed (L. E. Smith, 376); as well as a micro-colorimetric procedure (not yet published) suitable for the determination of the compound in spray residues; the increasing use of methyl bromide led to a method for its estimation in air in the small concentrations encountered in fumigation (Busbey and Drake, 400); and our studies of spray oils led to definite advances by Dawsey in methods of determining the deposits from oil emulsions (161, 292, 371) and of examining the oils for volatility (188).

The study of natural insecticides of plant origin and of synthetic organic insecticides has led to several improvements in micro and semi-micro methods of organic analysis. Improvements have been made by Clark in the procedures for determining carbon and hydrogen (197), nitrogen (203), halogens (227), sulfur (262), acetyl (316, 362), methoxyl and ethoxyl (131, 467), and a general article on micro analytical methods was presented before the Association of Official Agricultural Chemists by him (182), the present referee on that subject for this association.

Physical methods of analysis have also been given attention, and a new sedimentation method for the measurement of the particle-size distribution in insecticidal materials was developed (Goodhue and C. M. Smith, 315). The sedimentation and micro projection methods for determining the particle-size distribution of insecticides were compared, using three commercial samples of dusting sulfur. As far as precision is concerned, there is probably no choice between the methods, but the sedimentation method is less time-consuming (Goodhue and Gooden, 440).

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A process for the recovery of silver and iodine from silver iodide was worked out and patented (Spies, 241 and U. S. patent 2,060,539). In cooperation with Hillig of the Food and Drug Administration, Clark (416, 417) worked out a chemical procedure for evaluating spoilage in canned fish, especially salmon and tuna fish, using the improved apparatus and modified Dyer method to which reference has been made elsewhere. Directions were published by Clark for an improved method for the preparation of hydriodic acid suitable for alkoxyl and Friedrich-Kjeldahl nitrogen determinations (422).

The effect of particle size on the toxicity of calcium arsenate, paris green, synthetic cryolite, phenothiazine, and acid lead arsenate to the codling moth larva was studied by Goodhue in cooperation with Siegler (437). Coarse phenothiazine (particles having a midsize of 45 microns) was much less toxic than was the medium (15-micron) or fine (4-micron) fraction. No great difference in toxicity of the different fractions was obtained with the other insecticides.

APPLICATION OF STATISTICAL METHODS TO INSECTICIDE PROBLEMS

Much of the work on developing, testing, and establishing the commercial value of insecticides is necessarily of the variable nature which can be evaluated only by the application of the theory of probabilities. This is particularly true in the study of spray deposits and their removal by washing or other means, for the application of sprays is far from an exact procedure, the sprays are deposited on fruit with its natural large heterogeneity, and the nature of the washing technic employed is not accurately standardized. Further, in all tests against insects no amount of refinement in the mechanics of testing will overcome the inherent variations in susceptibility of individual insects, and recourse must be had to the statistical study of average effects.

The Division has long appreciated this situation and has continually striven to promote the statistical viewpoint in relation to insecticide problems; numerous publications have been issued calling the attention of other workers to the conclusions to be drawn from researches carried on in the Division.

Barnes (26) studied the distribution of arsenic residue on a large number of apples sprayed with lead arsenate, analyzing individual apples, and by the application of statistical considerations arrived at an estimate of the number of apples that must be used to insure adequate sampling. Barnes and Murray (40) extended this work to include washed apples, and again defined the size of representative samples. The results were obtained by the Gutzeit method, the variance of which was also determined by Barnes and Murray (48). Later, when methods of determining lead residues were developed to the point where their accuracy far exceeded that of the Gutzeit method, other lots of unwashed and washed apples were studied by Smith and Cassil (359) with regard to distribution and standard deviation of deposit, and still better estimates were arrived at regarding suitable sampling technic. These findings were then put into practice, so that Haller, Cassil, and Gould (325) were guided entirely by statistical considerations in estimating the variability of lead residues between trees in a plot, and the same authors together with Murray and Beaumont (398) critically judged their results of residue-removal studies on eastern

apples. Without the statistical background Fahey and Rusk (439) could not have intelligently interpreted their findings that the ratio between lead and arsenic in residues from lead arsenate does not change appreciably with time, and would not have been able definitely to show the variability of residues from part to part of an apple and from top to bottom of a tree (471). The statistical method has been carried by Spies (320) and Cassil and Wichmann (446) into our work on analytical methods. It also played an important part in some recent work (still unpublished) by Cassil on a survey of derris residues on cabbage, and in an investigation (also still unpublished) by McGovran and Cassil of the relation between the insecticidal potency of paris green and its degree of comminution.

NEW AND IMPROVED APPARATUS

Accomplishments of the Division of Insecticide Investigations under this heading include the development, in cooperation with entomologists, of an improved apparatus for the rapid vaporization of carbon disulfide (Weigel, Young, and Swenson, 4) and an improved burner for sulfur intended especially for mushroom-house fumigation with sulfur dioxide (Davis and Young, 237). Carbon disulfide is one of the most potent fumigants used for killing weevils in stored grains and cereals. The annual loss caused by these and other insects attacking these products is about $300,000,000. Carbon disulfide is also used for treating soil in greenhouses and for destroying Japanese beetle grubs in soil surrounding the roots of nursery stock. Several species of mushroom flies, manure flies, mushroom mites, and springtails cause an estimated annual reduction of about 20 percent in the potential value of the mushroom crop produced by commercial growers. This direct loss amounts to about $2,000,000 a year. One of the effective means of combating these pests is fumigation with sulfur dioxide. The development of improved means for applying these valuable fumigants assists in the fight against destructive pests.

In order accurately to study the effect of a fumigant upon a given insect, it is necessary to control very closely both the temperature and humidity. An improved equipment for laboratory fumigations with hydrocyanic acid which enables the operator to do this was developed and described (166). Special apparatus for studying the effect of a minute quantity of nicotine vapor upon aphids was devised (350) by Busbey in cooperation with H. H. Richardson. In the study of fumigants it is important to know their vapor pressures, because from these data can be calculated the amount of any fumigant necessary to saturate the atmosphere in a given space. An apparatus for measuring partial vapor pressures of binary systems was described by Nelson (135).

A new mechanical duster for applying insecticidal dusts was devised and patented by Goodhue (U. S. patent 2,114,092). The efficiency of an insecticide depends largely upon how thoroughly it is applied, and this new dusting device will enable the operator to apply nicotine dust and other dusts more effectively. Goodhue and Siegler (367) developed a motor-driven telescoping stirrer for use with an insecticide-spraying apparatus.

Many devices helpful to the chemist in his laboratory operations were invented or improved. These include an electrically heated sand bath (Clark, 198); a funnel-heating device (Markwood, U. S. patent 2,135,551 and 455); a differential-pressure control mechanism for vacuum distillation

which was developed by Palkin of the Bureau of Agricultural Chemistry and Engineering and Nelson of this Bureau (230); a new constant-temperature bath for heating vacuum stills (Nelson and Haller, 353); a sensitive thermoregulator for controlling the temperature of constant-temperature baths (Goodhue, 308 and U. S. patent 2,095,738); an improved melting-point apparatus (Schechter and Haller, 401); a laboratory spraying and washing apparatus (Chisholm, 450); and improved forms of apparatus for the determination of methoxyl groups (Clark, 131), for the identification of volatile fatty acids (Clark and Hillig, 416), for the determination of molecular weights (Spies, 162), and for collecting cigar smoke in order that its arsenic content may be determined (Gross and Nelson, 205).

In working with insecticides of plant origin, such as pyrethrum, tobacco, derris, and cube, it is necessary to extract the active principles by the use of an organic solvent. An improved large-capacity apparatus in which these extractions can be made was devised and described (Drake and Spies, 192), as was also (Schechter and Haller, 397) an automatic continuous percolator useful for the same purpose.

The particle size of insecticides has great influence on their efficacy, but heretofore no simple apparatus was known by which this could be determined. A new differential manometer type sedimentation apparatus was devised by Goodhue and C. M. Smith (315), by means of which the particle size of many insecticidal dusts may be accurately determined. A simpler apparatus for getting average particle size has recently been developed by Gooden and Smith (awaiting publication).

In order to determine arsenic, lead, copper, mercury, and other metals on fruits and vegetables and in timbers treated with termite-proofing materials or wood preservatives, it is necessary first to oxidize completely all the organic material present. An improved apparatus for accomplishing this oxidation was developed and described (Barnes, 49). A convenient portable rack for handling the glass flasks used in this operation was also described (Rusk, 313).

CHEMOTROPISM

The study of repellents for driving insects away from man and domestic animals or growing crops, and of attractants for luring them to poisoned baits or traps is embraced in the term chemotropism.

The work of the chemists of the Division of Insecticide Investigations in cooperation with Bishopp (1, 20, 123) and associates in this field has led to the use of pine-tar oil for repelling the screwworm fly and other blowflies from the wounds of cattle, sheep, goats, and other animals. This insect causes an estimated annual loss of nearly 5 million dollars in the United States, a loss which would be much greater except for the use of this remedy.

In addition to the development of a practical treatment for screwworms in livestock, these cooperative investigations brought to light information of theoretical value, such as the relation between the chemical constitution and physical properties of organic compounds and their repellent value against blowflies, the specificity of insecticides, the influence of volatility and other physical properties on repellent value, etc. This

information has proved helpful in the development of insecticides for the control of other insect pests.

Certain amyl esters of salicylic acid have been found to be useful as attractants for the tobacco hornworm moth by the Division of Truck Crop and Garden Insects Investigations. The preparation and physical properties of these were studied and described (Freeman and Haller, 408).

PHARMACOLOGICAL INVESTIGATIONS

The importance of ascertaining whether new synthetics and new principles isolated from insecticidal plants are deleterious to man was realized at the start of our work. Cooperative work was done in 1929 with Dr. D. E. Buckingham, a prominent veterinarian of Washington, D. C., in testing the action of rotenone upon dogs (Ind. Eng. Chem. 22:1133-1134. 1930). Later Dr. H. B. Haag, Professor of Pharmacology at the Medical College of Virginia, at Richmond, Va., was engaged as a collaborator and given the problem of studying the pharmacology of derris.

A report by Haag (119) on the toxicology of rotenone, one of the chief constituents of derris, cube, and timbo, was made in 1931. Later the pharmacological testing of insecticides was transferred to the Food Research Division of the Bureau of Agricultural Chemistry and Engineering, and further reports on derris and its constituents have issued from that Bureau (Ind. Eng. Chem. 28: 815-821. 1936; ibid. 29: 429-431. 1937; ibid. 30: 592-595. 1938). All this work was done at Richmond, Va., under the direction of Dr. Haag. Briefly it may be stated that, when taken by mouth, rotenone is only one-thirtieth as toxic as lead arsenate to rabbits, and there appears to be no danger of either acute or chronic poisoning from eating cabbage, etc., treated with derris or cube. Dr. Haag, while connected with the Division of Insecticide Investigations, also reported on the toxicology of anabasine (180), which closely resembles nicotine pharmacologically as well as physically, chemically, and insecticidally. At the request of the Division of Insecticide Investigations, pharmacological studies of phenothiazine and other synthetic insecticides have been carried out by the Bureau of Agricultural Chemistry and Engineering at San Francisco, Calif., but are not reported on here.

MOTHPROOFING

Clothes moths cause damage in the United States estimated at not less than $20,000,000 annually. Additional damage is caused by furniture beetles and carpet beetles attacking upholstered furniture, rugs, furs, wool, mohair, feathers, and other products containing keratin. Some authorities have figured the yearly damage caused by fabric pests plus the cost of insecticides, cedar closets, garment bags, etc., at not less than $100,000,000.

Accomplishments of the Division of Insecticide Investigations in cooperation with entomologists of the Bureau in this field include the discovery that rotenone is a mothproofing agent (Back, Cotton, and Roark, 85). Although exposure to direct sunlight and air decomposes rotenone into insecticidally inert substances, it does retain its remarkably high toxicity to clothes moth larvae under ordinary household conditions sufficiently long to enable it to be used in a practical way. This use of rotenone was also discovered by W. J. McGill (U. S. patent 1,854,948) and has been com-

mercially exploited by the Standard Oil Company of Indiana.

The literature of mothproofing, which is largely in patents, has been thoroughly abstracted, and these abstracts have been published by Roark from time to time (15, 44, 46, 107, 145). The three indexes of patented mothproofing materials (106, 170, 277) put out by Roark and Busbey constitute the most complete compilation of information on the subject and have been widely used by workers in this field.

TREE MEDICATION

The idea of introducing chemicals into the sap stream of a living plant in order to control diseases, insects, or fungi attacking the plant is an old one. Although this procedure has been somewhat discredited by the extravagant claims of quacks pretending to be tree doctors, beneficial results from the injection of certain compounds into trees have been reported. Preliminary to an investigation of this field of tree medication, the literature has been reviewed and a list made of the principal chemicals that have been proposed for this purpose (Carter, 433).

Several thousand analyses of sections of trees were made for the Division of Forest Insect Investigations to aid in tracing the flow of solutions of copper sulfate, sodium fluoride, zinc chloride, mercuric chloride, and other chemicals when injected into tree trunks for the control of bark beetles. New methods of analyzing impregnated wood were developed (Carter, Capen, and Cassil, 403).

ECONOMICS OF INSECTICIDES

This subject has received attention, as shown by the following publications by Roark:

Economic aspects of the use of insecticides (7)
United States insecticide statistics for 1928 (36)
The American market for tuba root (Derris elliptica) (77)
Problems in the production and use of insecticides in the South (78)
Insecticides and fungicides (248)
Insecticides and fungicides, 1918-1938 (394)

Although not complete, our compilation of statistics (394) of the consumption of insecticides and fungicides in the United States during 1936 is the best that has been published and has been widely quoted.

Additional compilations of figures on the use of certain insecticides have been prepared, as, for example, statistics of rotenone-bearing roots that are given in publications 159, 184, 279, and 419, also by Roark.

BIBLIOGRAPHIES, ABSTRACTS, AND REVIEWS

Information concerning insecticides is widely scattered, not only in the entomological and agricultural literature but also in the chemical, patent, pharmaceutical, medical, and veterinary literature. No one abstract journal covers all this literature, and it is exceedingly difficult for the student of insecticides to keep abreast of developments in this field. In order to assist in making information on insecticides available to those

interested in chemical methods of pest control, the Division of Insecticide Investigations has compiled and published a number of bibliographies on some of the more important insecticides, such as manganese arsenate (Dearborn, 337), magnesium arsenate (Dearborn, 411), spray residues of arsenic, lead, fluorine, nicotine, etc., and their removal (Busbey, 431), fluorine compounds (Carter and Busbey, 432), chloropicrin (79, 209, 253), ethylene dichloride (132), ethylene oxide (247), cyanide compounds (259, 281, 288, 470, 476), chemicals used in treatments of trees by injections (433), nicotine (306, 319), derris (134, 414, 421, 434), Lonchocarpus (279, 419), Tephrosia (327), Quassia (449), and miscellaneous insecticidal plants (94, 112).

The demand for these publications has been widespread, necessitating the reprinting of several.

ABSTRACTS OF PATENT LITERATURE

Beginning in 1926, abstracts of United States patents relating to insecticides, fungicides, spraying, dusting and fumigating machinery, insect traps, and other mechanical devices for combating pests were prepared by Roark and circulated in mimeographed form. At present these abstracts are issued monthly under the title "Review of United States Patents Relating to Pest Control," and sent to 1,235 persons in this country and to 232 in foreign countries. This service is greatly appreciated by economic entomologists, insecticide manufacturers, and pest-control operators. Many of the abstracts are reprinted by Soap, the official journal of the National Association of Insecticide and Disinfectant Manufacturers, and by Pests, the official organ of the National Pest Control Association. The British abstract journal Review of Applied Entomology also refers to this patent review.

From time to time the attention of entomologists has been called to the value of the information contained in patents. The following papers on this subject have been published by Roark:

Importance of patent literature to economic entomologists (3)
Insecticide patents (34)
The trend in pest-control inventions in the United States (147)
A review of nonchemical United States patents issued from 1917 to 1933, inclusive, relating to insect control (254)

Comprehensive compilations of information taken from the patent literature concerning certain classes of insecticides have been issued. The most complete of these relate to mothproofing (15, 44, 46, 106, 107, 145, 170, 277), to organic sulfur compounds used as insecticides and fungicides (250), and to the rotenone-bearing plants Derris, Lonchocarpus, and Tephrosia (76, 134, 211, 279, 327, 409, 419).

There were also issued by Roark 47 mimeographed lists of United States patents issued from 1917 to 1933, inclusive, relating to insect electrocutors, traps, fly screens, fumigating devices, dusting and spraying apparatus, insect barriers, machinery for washing insecticide spray residues from fruits and vegetables, etc. These lists, which briefly abstracted 3,543 patents on these subjects, were distributed to State entomologists and plant pathologists and others interested in pest control, and constitute

the only comprehensive publication on nonchemical means of pest control other than the original patents filed in the United States Patent Office.

An index of chemicals mentioned in the Review of United States Patents relating to Pest Control issued in 1931 was published by Roark (200).

BOOK REVIEWS

Because of their reputation as outstanding specialists in certain fields of research, members of the Division of Insecticide Investigations have been asked from time to time, by the editors of leading scientific journals such as the Journal of the Association of Official Agricultural Chemists, Industrial and Engineering Chemistry, and Cereal Chemistry, to prepare reviews of books on pyrethrum (Roark, 206), organic chemistry (Haller, 280), theoretical chemistry (Nelson, 314), qualitative analysis (Nelson, 360), fumigation (Roark, 375*, microchemical methods (Clark, 418), and insecticide statistics (Roark, 469).

POPULAR ARTICLES ON INSECTICIDES

Many popular articles presenting information on insecticides have been published. The following titles indicate the scope of our activities under this heading:

Modern methods of disinfecting and fumigating (5)
A report of the Insecticide Division's investigations with various insecticides (9, 10)
Chemists in front ranks in warfare on harmful insects (12)
New insecticides developed to improve crop protection (50)
Some of the chemical problems in codling moth control (59)
Use of economic poisons to safeguard crops (114)
New fumigants for destroying insect pests in foodstuffs (116)
Rotenone, a promising new insecticide (133)
Chemically combating insect pests of foodstuffs (143)
Advantages and limitations of organic insecticides (239)
Recent developments in insecticide research (240)
Insecticides for combating household pests (271)
Research to find substitutes for lead arsenate (300)
An insect that breathes through its nose (345)
Agricultural products as insecticides (429)
Recent progress in the chemistry of pyrethrum flowers (443)

The difference between insecticides and fungicides was pointed out in a brief note (392) and the term "derris resinate" criticized (404).

PAPERS PREPARED BY INVITATION

By invitation of the National Research Council, a series of annual reviews of the progress in insecticides and fungicides in the United States was prepared by Roark and published in the Annual Survey of American Chemistry for 1929, 1930, 1931, 1933, and 1935 (57, 105, 140, 218, 293). Each of these reviews summarizes the information from about 200 to 350 scientific articles and patents. Also, by invitation, progress in the chemistry of heterocyclic compounds recorded by American workers in 1931 (139) and 1932 (181) was reviewed and published by Haller and L. E. Smith in the Annual Surveys. By invitation from the editors of the Annual Review of Biochemistry, a chapter on developments during 1937 in organic insecticides of plant origin

was prepared by LaForge and Markwood for volume 7 of this work (393)

The literature and patents describing progress from June to November, 1938, in household insecticides, especially pyrethrum, derris, and synthetics, was reviewed at the request of the National Association of Insecticide and Disinfectant Manufacturers (Roark, 426).

Other papers prepared by invitation include Nos. 5, 7, 9, 10, 42, 50, 58, 78, 90, 114, 477. These are discussed under the subjects of which they treat, such as pyrethrum, derris, etc.

LIST OF PUBLICATIONS AND PATENTS

of the

DIVISION OF INSECTICIDE INVESTIGATIONS

R. C. Roark, Principal Chemist in Charge

for the period

July 1, 1927, to December 31, 1939

Some of the publications listed below were prepared jointly by members of the Division of Insecticide Investigations and those of other organizations. The asterisk indicates the authors not connected with this Division. Copies of these publications can doubtless be consulted in almost any large public or institutional library.

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6. Fumigation Tests with Certain Aliphatic Chlorides. R. C. Roark and R. T. Cotton.* Jour. Econ. Ent., v. 21, no. 1, p. 135-142, Feb. 1928.

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Index
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Index
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Index
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Index
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Index
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Index
No.

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Index
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Index No.

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Index
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Index No.

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Index
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Index
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Index
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Index No.

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Index
No.

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Index
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Index
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Index
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Index
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Index
No.

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Index
No.

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Index
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Index
No.

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Index
No.

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Index
No.

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Index
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Index
No.

479. A Method for Determining Deguelin in Derris and Cube. L. D. Goodhue and H. L. Haller. Indus. Engin. Chem., Anal. Ed., vol. 11, no. 12, pp. 640-642. December 15, 1939.

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PATENTS OF THE DIVISION OF INSECTICIDE INVESTIGATIONS[1]

Index
No.

1. 1,789,322 (Jan. 20, 1931; appl. Nov. 23, 1927). MATERIAL FOR KILLING INSECTS. R. C. Roark and R. T. Cotton.* (Chloroacetates as fumigants.)

2. 1,791,429 (Feb. 3, 1931; appl. Feb. 23, 1929). INSECTICIDE AND FUMIGANT. R. C. Roark and R. T. Cotton.* (Ethylene oxide as an insecticidal fumigant.)

3. 1,842,443 (Jan. 26, 1932; appl. Nov. 15, 1929). PROCESS FOR THE MANUFACTURE OF INSECTICIDES AND METHOD OF MAKING SAME. R. H. Carter. (Manufacture of double fluorides of the alkali metals with aluminum by treatment of a water insoluble compound of aluminum with alkali metal compounds and hydrofluoric acid.)

4. 1,863,266 (June 14, 1932; appl. Nov. 15, 1929). PROCESS FOR THE MANUFACTURE OF INSECTICIDES AND METHOD OF MAKING SAME. R. H. Carter. (Manufacture of double fluorides of the alkali metals with aluminum in admixture with hydrated silica as diluent by treatment of water soluble salts of aluminum with alkali metal compounds and hydrofluosilicic acid.)

5. 1,863,519 (June 14, 1932; appl. Nov.15, 1929). PROCESS FOR THE MANUFACTURE OF INSECTICIDES AND METHOD OF MAKING SAME. R. H. Carter. (Double fluorides of the alkali metals with aluminum in admixture with hydrated alumina as diluent by treatment of water soluble salts of aluminum with alkali metal compounds and hydrofluoric acid.)

6. 1,884,966 (Oct. 25, 1932; appl. Feb. 19, 1930). PROCESS FOR THE REMOVAL OF NATURAL OILS, WAX, AND SPRAY RESIDUES FROM FRUITS. R. H. Robinson. (With hydrochloric acid, water, and a petroleum hydrocarbon.)

7. 1,885,100 (Oct. 25, 1932; appl. July 11, 1929). PROCESS FOR THE REMOVAL OF SPRAY RESIDUES FROM FRUITS AND VEGETABLES. R. H. Robinson. (By means of a mixture of hydrochloric acid and ferrous sulphate.)

8. 1,928,256 (Sept. 26, 1933; appl. Aug. 23, 1930). INSECTICIDE AND PROCESS OF MAKING THE SAME. H. A. Jones. (Colloidal dispersions of rotenone by the aid of pyridine.)

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[1] Copies of all patents can be obtained for 10 cents (no postage) sent to the Patent Office, Washington, D. C. In ordering a copy of a patent the number of the patent, the date, the name of the patentee, and the subject of the invention should be stated.

Index
No.

9. 1,923,968 (Oct. 3, 1933; appl. Aug. 23, 1930). PROCESS FOR MAKING AN INSECTICIDE. H. A. Jones. (Colloidal dispersions of rotenone by the aid of tannic acid.)

10. 1,933,975 (Nov. 7, 1933; appl. May 29, 1933.) PROCESS OF PREPARING ACYLATED PHENOLS. H. L. J. Haller and P. S. Schaffer.

11. 1,942,104 (Jan. 2, 1934; appl. Feb. 20, 1933). PROCESS OF EXTRACTING ROTENONE FROM PLANT MATERIAL. H. A. Jones. (By means of carbon tetrachloride.)

12. 1,945,312 (Jan.30, 1934; appl. Apr. 10, 1933). PROCESS OF PREPARING DIHYDROROTENONE. H. L. J. Haller and P. S. Schaffer.

13. 2,024,027 (Dec. 10, 1935; appl. Apr. 10, 1931). FUMIGANT. R. T. Cotton* and H. D. Young. (Ethylene oxide-carbon dioxide mixture.)

14. 2,033,853 (March 10, 1936; appl. July 5, 1934). COMPOUNDS OF BENTONITE WITH ORGANIC BASES AND PROCESS OF PRODUCING SAME. C. R. Smith.

15. 2,044,502 (June 16, 1936; appl. Feb. 18, 1936). PROCESS FOR THE PURIFICATION OF PYRETHRIN CONCENTRATES. H. L. J. Haller and F. B. LaForge.

16. 2,046,546 (July 7, 1936; appl. May 23, 1934). PROCESS FOR THE REMOVAL OF INSECTICIDAL RESIDUES FROM FRUITS AND VEGETABLES. R. H. Carter. (Aluminum salts with and without dilute acids.)

17. 2,046,547 (July 7, 1936; appl. May 23, 1934). PROCESS FOR THE REMOVAL OF INSECTICIDAL RESIDUES FROM FRUITS AND VEGETABLES. R. H. Carter. (Ferric salts with and without dilute acids.)

18. 2,046,548 (July 7, 1936; appl. May 23, 1934). PROCESS FOR THE REMOVAL OF INSECTICIDAL RESIDUES FROM FRUITS AND VEGETABLES. R. H. Carter and J. E. Fahey. (Boric acid with dilute mineral acids.)

19. 2,049,725 (Aug. 4, 1936; appl. March 20, 1935). INSECTICIDE. L. E. Smith. (Diaryl thioxins, especially phenothioxin.)

20. 2,050.974 (Aug. 11, 1936; appl. Sept. 12, 1935). PROCESS OF PREPARING A PURIFIED PYRETHRIN CONCENTRATE. F. B. LaForge and H. L. J. Haller.

21. 2,060,539 (Nov. 10, 1936; appl. May 21, 1935). PROCESS FOR OBTAINING FREE SILVER AND IODINE FROM SILVER IODIDE. J. R. Spies.

22. 2,063,941 (Jan. 5, 1937; appl. July 9, 1935). INSECTICIDE AND PROCESS FOR MAKING THE SAME. L. N. Markwood. (Nicotine humate.)

Index
No.

23. 2,030,004 (May 11, 1937; appl. May 17, 1933). DOUBLE SALT OF COPPER ARSENITE AND A COPPER SALT OF AN UNSATURATED FATTY ACID AND PROCESS OF MAKING THE SAME. F. E. Dearborn.

24. 2,094,831 (Oct. 5, 1937; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [Covers the use as insecticides of hydroxy azo compounds containing at least one hydroxy group and at least one nitro group; for example, 4-(p-nitrophenylazo)-resorcinol and 1-(p-nitrophenylazo)-2-naphthol.]

25. 2,095,738 (Oct. 12, 1937; appl. Jan. 29, 1937). THERMOREGULATOR. L. D. Goodhue.

26. 2,095,939 (Oct. 12, 1937; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises an aryl hydroxy azo compound in which the nuclei are substituted by one azo, one or more hydroxy, and one or more halogen groups; for example, p-(p-bromophenylazo)-phenol. 4-(p-bromophenylazo)-resorcinol, and 4-(2,5-dichlorophenylazo)-phenol.]

27. 2,095,940 (Oct. 12, 1937; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises an arylazo compound in which the nuclei are substituted by at least one halogen atom; for example, p-iodoazobenzene.]

28. 2,095,941 (Oct. 12, 1937; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises a compound containing homocyclic nuclei joined by one azo group, and containing in addition to not less than one hydroxyl group, not less than one halogen atom and not less than one alkyl group; for example, 4-(2,5-dichlorophenylazo)-o-cresol, 4-(p-bromophenylazo)-o-cresol, and 4-(p-bromophenylazo)-m-cresol.]

29. 2,096.414 (Oct. 19, 1937; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises a compound selected from the group consisting of 4-phenylazo-o-toluidine, 4-phenylazo-m-toluidine, p-(o-tolylazo)-aniline, p-(m-tolylazo)-aniline, p-(p-tolylazo)-aniline, 1-(o-tolylazo)-2-naphthylamine, 4-(o-tolylazo)1-naphthylamine, 1-(m-tolylazo)-2-naphthylamine, 1-(p-tolylazo)-2-naphthylamine, 4-(m-tolylazo)-1-naphthylamine, 4-(p-tolylazo)-1-naphthylamine, and 4-(o-tolylazo)-o-toluidine.]

30. 2,096,566 (Oct. 19, 1937; appl. Dec. 27, 1933). COMPOUND AND PROCESS FOR MAKING SAME. C. R. Smith. [An insecticide comprising nicotine-bentonite compounds.]

Index
No.

31. 2,096,715 (Oct. 26, 1937; appl. Feb. 26, 1937). PROCESS OF PREPARING DIHYDROJASMONE. H. L. Haller and F. B. LaForge.

32. 2,099,826 (Nov. 23, 1937; appl. July 22, 1937). INSECTICIDE. P. S. Schaffer and H. L. Haller. [Dialkylacridan.]

33. 2,100,493 (Nov. 30, 1937; appl. June 3, 1936). INSECTICIDE. L. E. Smith and H. V. Claborn. [o-, m-, and p-Nitroiodobenzenes.]

34. 2,103,195 (Dec. 21, 1937; appl. July 3, 1937). INSECTICIDE. H. A. Jones. [Combination of rotenone and dichloroacetic acid.]

35. 2,104,584 (Jan. 4, 1938; appl. Oct. 15, 1934). DOUBLE SALTS OF COPPER ARSENITE AND A COPPER SALT OF A HIGHER UNSATURATED FATTY ACID. F. E. Dearborn. [Compounds containing arsenic, copper and the anyhdride of a fatty acid of the general formulae CnH_2n-4O_2 and CnH_2n-6O_2.]

36. 2,107,058 (Feb. 1, 1938; refiled for abandoned appl. Serial No. 744,636, Sept. 19, 1934. This application June 24, 1937, Serial No. 150,165). PROCESS FOR MAKING AN INSECTICIDE. L. N. Markwood. [Nicotine peat.]

37. 2,110,614 (March 8, 1938; appl. May 10, 1937). INSECTICIDE. D. L. Vivian and H. L. Haller. [Phenazine.]

38. 2,110,896 (March 15, 1938; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises a compound of the general formula $CH_3 - R_1 - N = N - R_2 - OH$ where R_1 and R_2 denote interchangeable homocyclic aryl nuclei, R_1 being a single benzene ring and R_2 a naphthyl nucleus, for example, 1-(o-tolylazo)-2-naphthol and 4-(o-tolylazo)-1-naphthol.]

39. 2,110,897 (March 15, 1938; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises a compound of the general formula $R - N = N - R_1 - (OH)$ where R and R_1 denote interchangeable homocyclic aryl nuclei, R being a single benzene ring and R_1 a naphthyl nucleus, for example, 4-phenylazo-1-naphthol, (1-naphthylazo)- phenol, and 4-(2-naphthylazo)-1-naphthol.]

40. 2,111,879 (March 22, 1938; appl. June 27, 1936). INSECTICIDE. D. L. Vivian and H. L. Haller. [This insecticide comprises a compound of the general formula $R_1 - N = N - R_2 - (NH_2)$ where R_1 and R_2 denote interchangeable aryl nuclei, R_1 being a single benzene nucleus and R_2 a naphthyl nucleus, for example, 1-phenylazo-2-naphthylamine, p-(1-naphthylazo)-aniline, and 1-(1-naphthylazo)-2-naphthylamine.]

Index
No.

41. 2,114,092 (Apr. 12, 1938; appl. Mar. 9, 1937). MECHANICAL DUSTER. L. D. Goodhue.

42. 2,115,046 (Apr. 26, 1938; appl. Feb. 26, 1937). INSECTICIDE. L. E. Smith. [4,6-Dinitro-o-tolyl methyl ether.]

43. 2,123,248 (July 12, 1938; appl. Jan. 14, 1937). INSOLUBLE REACTION PRODUCTS OF PEAT AND PROCESS OF MAKING SAME. L. N. Markwood. [Water-insoluble reaction products of peat with organic bases of the group consisting of arecoline, anabasine, codeine, ethylene diamine and piperidine.]

44. 2,127,090 (Aug. 16, 1938; appl. Feb. 26, 1937). INSECTICIDE. L. E. Smith. [4,6-Dinitro-o-tolyl acetate]

45. 2,127,380 (Aug. 16, 1938; appl. Apr. 14, 1937). INSECTICIDE AND FUNGICIDE. Frederick E. Dearborn. [Compounds of copper, arsenic, and unsaturated acids]

46. 2,129,517 (Sept. 6, 1938; appl. Jan. 10, 1938). ADHESIVE FOR INSECTICIDES AND A PROCESS FOR MAKING SAME. Lyle D. Goodhue. (Pine-oil foots)

47. 2,135,551 (Nov. 8, 1938; appl. Sept. 24, 1936). FUNNEL HEATING DEVICE. Louis N. Markwood.

48. 2,140,481 (December 13, 1938; appl. Jan. 28, 1938). INSECTICIDE. William G. Rose and Herbert L. J. Haller. [Pentaerythrityl bromide.]

49. 2,146,257 (Feb. 7, 1939; appl. Sept. 23, 1938). ADHESIVE FOR INSECTICIDES. L. D. Goodhue. [Furfural with amines.]

50. 2,146,258 (Feb. 7, 1939; appl. Sept. 26, 1938). ADHESIVE FOR INSECTICIDES. L. D. Goodhue. [Furfural with ketones.]

51. 2,149,303 (March 7, 1939; appl. Oct. 13, 1938). LIQUID MEASURING DEVICE. L. N. Markwood.

52. 2,150,759 (March 14, 1939; appl. Jan. 28, 1938). INSECTICIDAL AND FUNGICIDAL COMPOSITIONS. R. H. Carter. [Cuprous amyl xanthate and diamyl xanthogen composition.]

53. 2,155,010 (April 18, 1939; appl. April 1, 1938). INSECTICIDE. Andrew F. Freeman. [N-nitrosodiphenylamine.]

54. 2,159,585 (May 23, 1939; appl. Jan. 28, 1938). COMPOUNDS OF COPPER, ARSENIC, AND A SULPHMONOCARBOXYLIC ACID AND PROCESSES OF MAKING THEM. F. E. Dearborn.

Index
No.

55. 2,163,000 (June 20, 1939; appl. June 14, 1938). SEDIMENTATION APPARATUS. L. D. Goodhue and C. M. Smith.

56. 2,169,793 (Aug. 15, 1939; appl. Feb. 11, 1937). PROCESSES OF TREATING GLYCERIDES. F. E. Dearborn.

57. 2,173,333 (Sept. 19, 1939; appl. Aug. 16, 1937). COPPER XANTHATE. R. H. Carter. [Ethylene glycol]

58. 2,173,384 (Sept. 19, 1939; appl. Aug. 16, 1937). COPPER XANTHATE. R. H. Carter. [Diethylene glycol]

59. 2,173,386 (Sept. 19, 1939; appl. Feb. 6, 1939). INSECTICIDE. D. L. Vivian. [Hydrazobenzene]

60. 2,175,109 (Oct. 3, 1939; appl. July 22, 1937). INSECTICIDE. H. V. Claborn and L. E. Smith. [9-Chlorofluorene.]

AUTHOR INDEX

(The asterisk indicates that the author is not a member of the Division of Insecticide Investigations. The letter "P" refers to the list of patents which follows the list of publications. Numbers used here are the index numbers of the publications and patents listed.)

Anon.....269
*Abbott, W. S..... 66
Acree, F., Jr.....220, 263, 297, 302, 370, 478, 481
*Back, E. A..... 64, 85
Barnes, J. W..... 26, 40, 48, 49
*Beaumont, J. H.....242, 252, 398
*Bishopp, F. C..... 1, 20, 123
*Bulger, J. W.....474
Busbey, R. L.....148, 247, 250, 253, 277, 306, 319, 350, 400, 431, 432, 449, 476
*Campbell, F. L.....177, 178, 210, 215, 233, 245, 264
Capen, R. G.....403
Carter, R. H..... 21, 22, 38, 55, 67, 68, 97, 100, 102, 146, 156, 157, 158, 186, 194, 195, 201, 212, 225, 226, 267, 311, 354, 403, 432, 433, 463
P 3, P 4, P 5, P 16, P 17, P 18, P 52, P 57, P 58
Cassidy, John.....278, 465
Cassil, C. C.....252, 305, 325, 338, 344, 359, 386, 390, 391, 398, 403, 444, 446.
Chisholm, R. D.....420, 442, 450
Claborn, H. V.....155, 272, 339, 381, 388
P 33, P 60
Clark, E. P..... 41, 56, 60, 87, 89, 92, 104, 108, 111, 118, 131, 136, 144, 151, 155, 163, 171, 175, 182, 197, 198, 203, 214, 227, 255, 262, 290, 310, 316, 333, 362, 365, 389, 416, 417, 418, 422, 456, 467
*Clifford, P. A.....208
*Cotton, R. T..... 2, 6, 8, 11, 16, 30, 54, 64, 85, 273, 294, 309, 336, 346, 358, 361, 428
P 1, P 2 P 13
*Cox, J. H..... 64
*Cressman, A. W.....224, 299, 330
Cupples, H. L.....166, 167, 172, 259, 266, 275, 281, 283, 284, 288, 298, 349, 379, 383, 468. 472
*Davidson, J.....391
*Davidson, W. M..... 43, 66, 95, 96
*Davis, A. C.....216, 237, 246, 272
Dawsey, L. H.....161, 188, 224, 292, 299, 330, 371
Dearborn, F. E..... 63, 71, 260, 235, 324, 337, 357, 369, 411
P 23, P 35, P 45, P 54, P 56
*Diehl, H. C..... 72, 81, 142
Drake, N. L..... 75, 130, 148, 149, 192, 236, 243, 400
Fahey, J. E..... 59, 72, 81, 142, 153, 183, 212, 256, 301, 348, 384, 402, 405, 407, 439, 447, 451, 471, 475
P 18

*Fink, D. E. 296, 303, 304, 331, 381
Freeman, A. F. 408
P 53
Gersdorff, W. A. 43, 70, 79, 86, 103, 132, 174, 177, 189, 213, 257, 258, 270, 323, 335, 388, 415, 460
Gertler, S. I. 425
Gooden, E. L. 177, 274, 332, 368, 440
Goodhue, L. D. 276, 308, 315, 343, 367, 368, 374, 386, 396, 420, 436, 437, 440, 457, 479, 430
P 25, P 41, P 46, P 49, P 50, P 55
*Gould, Edwin 325, 398
*Graham, J. J. T. 373, 377
Gross, C. R. 59, 168, 196, 205, 212, 219, 242, 251
Haag, H. B. 119, 180
*Haas, A. J. 161
Haller, H. L. 61, 69, 82, 93, 109, 110, 117, 124, 126, 129, 137, 138, 139, 160, 179, 181, 190, 193, 199, 204, 207, 220, 223, 228, 231, 233, 263, 265, 280, 282, 291, 296, 297, 307, 318, 326, 328, 329, 331, 339, 340, 341, 353, 355, 382, 385, 397, 399, 401, 406, 408, 413, 424, 425, 430, 436, 443, 453, 457, 458, 479, 480
P 10, P 12, P 15, P 20, P 24, P 26, P 27, P 28, P 29, P 31, P 32, P 37, P 38, P 39. P 40, P 48
*Haller, M. H. 242, 252, 325, 398
*Haring, M. M. 363
*Harris, M. 208
Hiley, J. 284, 298, 330, 371
*Hillig, F. 416, 417
Jones, H. A. 62, 95, 96, 101, 110, 113, 122, 133, 164, 165, 169, 176, 177, 202, 210, 215, 217, 245, 264, 289, 322, 334, 352, 366, 373, 377, 380, 395, 423, 441, 459, 464
P 8, P 9, P 11, P 34
*Keenan, G. L. 66, 94, 125, 163, 228
Koblitsky, L. 462
*Laake, E. W. 1, 20, 123
LaForge, F. B. 17, 18, 19, 31, 35, 51, 52, 53, 61, 65, 69, 74, 82, 83, 109, 115, 117, 120, 124, 125, 126, 129, 137, 150, 152, 179, 191, 223, 231, 232, 265, 282, 291, 302, 307, 328, 329, 340, 341, 355, 370, 393, 399, 430, 443, 478, 481
P 15, P 20, P 31
*Lathrop, F. H. 284
*Leukel, R. W. 364, 473
*Loughrey, J. H. 208
Love, S. 366
Markwood, L. N. 236, 287, 351, 378, 393, 410, 445, 455
P 22, P 36, P 43, P 47, P 51
*McDonnell, C. C. 66
*McGovran, E. R. 406

*McIndoo, N. E.....306, 319
*Munger, F.....261, 295, 312, 387, 435, 461
Murray, C. W..... 40, 48, 91, 141, 154, 208, 212, 252, 278, 398, 454, 466
Nelson, O. A..... 14, 23, 24, 27, 29, 66, 73, 84, 128, 135, 185, 205, 229, 230, 314, 344, 353, 360, 363, 364, 452, 473, 474
*Newcomer, E. J.....100, 186, 194, 201
*Palkin, S.....230
*Parman, D. C..... 1, 20, 123
*Phillips, G. L.....406
*Pulley, G. N.....391
*Reeves, E. L.....466
*Richardson, C. H..... 80
*Richardson, H. H.....350
Roark, R. C..... 1, 2, 3, 5, 6, 7, 8, 9, 10, 11, 12, 15, 16, 20, 21, 25, 28, 29, 32, 34, 36, 42, 44, 46, 50, 54, 57, 58, 76, 77, 78, 84, 85, 90, 94, 98, 99, 105, 106, 107, 112, 114, 116, 123, 134, 140, 143, 145, 147, 159, 170, 184, 187, 200, 206, 209, 211, 218, 221, 238, 239, 240, 244, 248, 250, 253, 254, 271, 277, 279, 293, 300, 319, 327, 345, 372, 375, 394, 404, 409, 414, 419, 421, 426, 427, 429, 434, 438, 448, 469, 477
P 1, P 2
Robinson, R. H..... 39
P 6, P 7
Rose, W. G.....318, 382
P 48
Rusk, H. W.....242, 301, 313, 347, 348, 384, 402, 407, 439, 471, 475
*Ryall, A. L..... 72, 153, 212, 278, 454
*Sazama, R. F.....256, 301, 384, 402, 405, 407, 475
Schaffer, P. S.....193, 199, 204, 297, 331, 458
P 10, P 12, P 32
Schechter, M. S.....397, 401, 413, 453
*Shepard, H. H..... 80, 195
*Siegler, E. H.....261, 295, 312, 367, 387, 435, 437, 461
Smith, C. M..... 62, 91, 169, 219, 268, 274, 289, 305, 315, 332, 359
P 55
Smith, C. R..... 13, 33, 47, 80, 88, 121, 127, 178, 222, 249, 356
P 14, P 30
*Smith, E.....212, 278, 465
Smith, L. E..... 35, 51, 52, 65, 74, 83, 115, 124, 139, 150, 179, 181, 207, 232, 233, 261, 295, 304, 312, 321, 376, 381, 387, 392, 412, 424, 435, 461
P 19, P 33, P 42, P 44, P 60
Spies, J. R..... 75, 130, 149, 162, 173, 192, 234, 235, 236, 241, 243, 317, 320, 342
P 21

*Steiner, L. F.....256, 301, 384, 402, 405, 407, 447, 451, 475
*Sullivan, W. N.....177, 178, 210, 215, 233, 245, 264, 352, 385, 395, 406, 457
*Summerland, S. A.....447
*Swenson, R. L..... 4
Vivian, D. L.....303, 381
P 24, P 26, P 27, P 28, P 29, P 37, P 38, P 39, P 40, P 59
*Vorhes, F. A., Jr.....208
*Wagner, G. B.....273, 294, 309, 336, 346, 358, 361, 428
*Weigel, C. A..... 4
*Wichmann, H. J.....208, 446
*Yothers, M. A.....466
Young, H. D..... 4, 27, 30, 37, 45, 64, 128, 185, 216, 237, 246, 247, 273, 294, 309, 336, 346, 358, 361, 428, 470
476
P 13
*Yust, H. R.....284, 298

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